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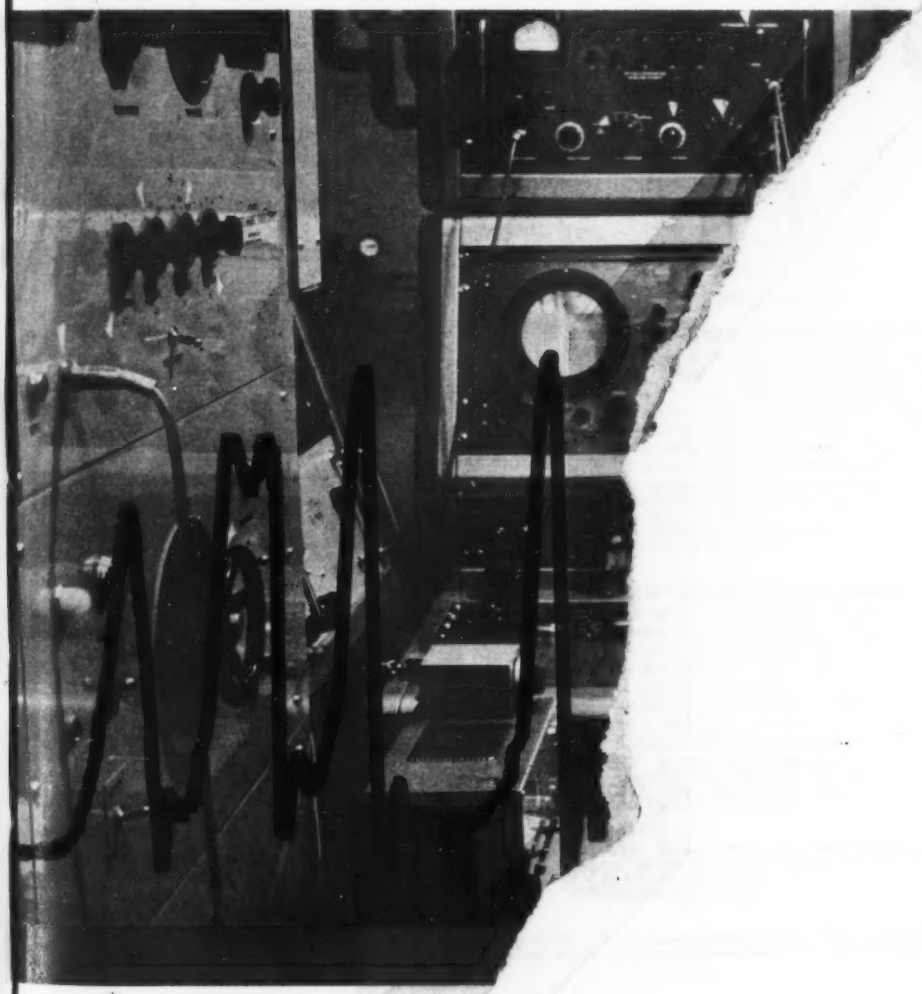

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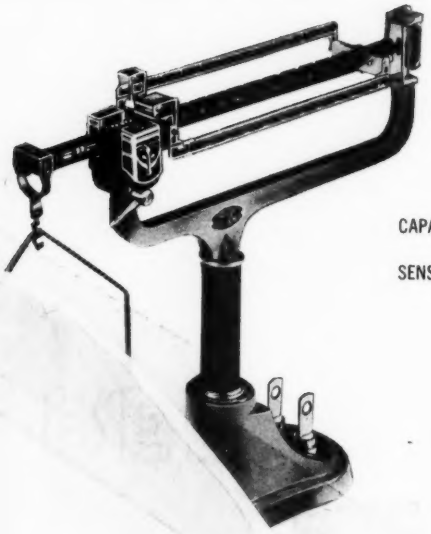

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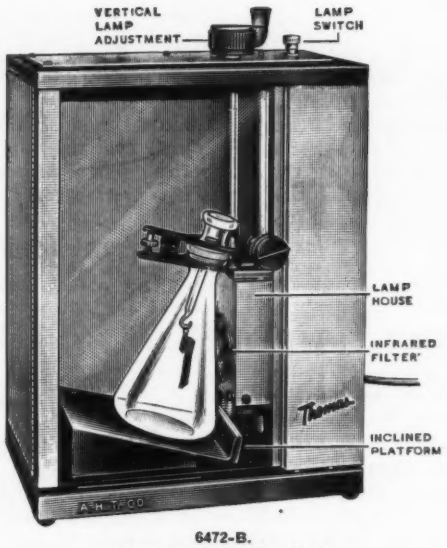
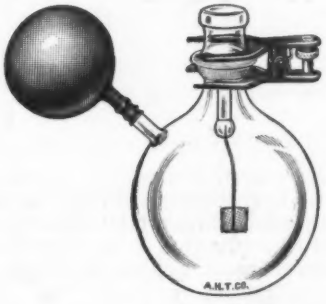


6472-B.
Showing 6471-P10 Ogg Flask in position *(Flask not included)*

6471-P10.



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*Patent applied for. Based on developments by Clyde L. Ogg and associates, Eastern Utilization Research and Development Division, A.R.S., U.S.D.A., Philadelphia, Pa. Described at the International Symposium on Microchemical Techniques, August, 1961.

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Chemical Instrumentation

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. . . a Chem Ed feature

This series of articles presents a survey of the basic principles, characteristics, and limitations of those instruments which find important applications in chemical work. The emphasis is on commercially available equipment, and approximate prices are quoted to show the order of magnitude of cost of the various types of design and construction.

14. Chromatographic Equipment (Concluded)

The effects of solvent evaporation from the free surfaces of a paper or gel electrophoresis support due to the heating produced by the flow of current can be eliminated by clamping the sheet or bed between glass or plastic plates. This approach to supported electrophoresis has been adopted in the instrumentation designed by E. C. Apparatus Co., Swarthmore, Pennsylvania. Figure 48 shows a

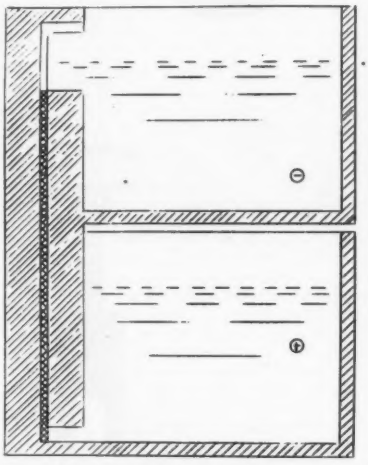

Figure 48. Diagram of the EC Apparatus Co. Vertical Gel Electrophoresis Apparatus. The upper and lower electrode compartments are shown; buffer solution contacts the upper and lower ends of the gel block shown in the annular channel between plastic confining plates. These plates are cooled by imbedded water coils (not shown).

schematic diagram of the EC470 Vertical Gel Electrophoresis Apparatus ($325), and Figure 49 shows the actual appearance of the instrument. In this device, which is intended primarily for use with gels, the medium is contained between non-wetting, non-conductive plastic surfaces; these surfaces are the bounding planes of structures which contain imbedded tubing through which cooling water continually circulates. This arrangement prevents evaporation by (1) providing a physical barrier to the escape of vapor molecules, and (2) conducting electrical heat away as rapidly as it is generated. In several of the paper electrophoresis devices previously described, the paper rests on a water-cooled table for removal of the electrical heat. The E.C. apparatus differs in that the water-cooled surface completely surrounds the electrophoresis medium.

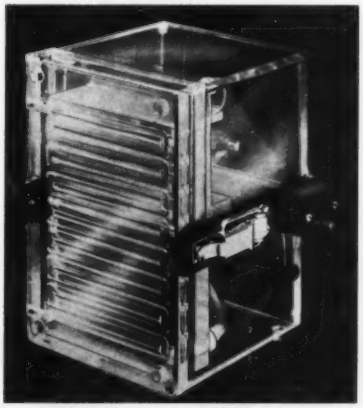

Figure 49. Photograph of Model EC470 Vertical Gel Electrophoresis Apparatus. The cooling coils imbedded in the plastic confining plates referred to in Figure 48 can be seen.

Because the medium is supported on both sides, this apparatus can be used equally well in the vertical and horizontal positions. The buffer compartments are physically separate, and electrical communication between the electrodes in these chambers can only occur along the path provided by the electrophoresis medium. The electrodes consist of platinum wires stretched between opposing walls in the buffer compartments.

This company also makes an apparatus designed for use with either paper or a gel (Model EC455, complete with 1000 v, 200 ma power supply, $545). If paper is to be used, it is placed between plastic insulating shields, a rubber pad is added, and the combination is compressed between water cooled plastic plates.

High Voltage Pherographs

Electrophoresis with high potential gradients applied to the medium has the advantage that the electromigration can be made to occur with sufficient rapidity that the separation of two components having slightly different mobilities is not disturbed by the random diffusional spreading out that occurs when prolonged runs are necessary. Thus, in many cases the sharpness of separations can be markedly improved by operating at high applied voltages. However, the success of such procedures depends largely upon the effectiveness of the means employed for conducting away the heat generated by the passage of current. Since the power dissipated by the electricity increases as the square of the voltage at constant resistance ($P = E^2/R$), the efficiency of the cooling is of critical importance in this type of instrument.

Two instruments specifically designed for high voltage paper electrophoresis are made by Gilson Medical Electronics, Middleton, Wisconsin. The Model M Electrophorator ($4800) contains a refrigerated electrophoretic platform, cooled by the fluid circulated from a 1-hp refrigerator. A glass plate covered with a thin Teflon membrane lies on this refrigerated platform, and the paper strips or sheet lie on the Teflon. The chamber cover has a polyethylene membrane mounted below its lower surface, with an air space between the membrane and the cover. If air pressure is applied, the polyethylene is pressed against the paper strip, uniformly compressing it against the refrigerated Teflon surface, and insuring good cooling. If the air pressure is reduced below atmospheric pressure, the polyethylene membrane is sucked up, forming a shallow chamber above the paper; this is done whenever it is necessary to facilitate moisture equilibration. The power supply delivers up to 20,000 v and 50 ma. The GME Model D electrophorator ($1950) employs a power supply that delivers up to 5000 v at 300 ma. Filter paper sheets up to 18″ × 22″ are immersed in a fiberglass tank containing a light petroleum oil (Varsol, available from Standard Oil Company of New Jersey). Stainless steel coils through which cold tap water circulates are mounted in the oil at the top of the tank, and serve to remove the heat transmitted to the oil from the paper.

A high voltage paper electrophoresis unit is also available from Shandon Scientific Co., London (U.S. distributor: Consolidated Laboratories, Inc., Chicago Heights, Illinois; price: about $2100). The power supply delivers up to 10,000 v at 100 ma. The paper is sandwiched between polyethylene sheets, and upper and lower watercooled platens are pressed against both sides of this sandwich by the application of air pressure.

Column Electrophoresis

Instruments designed for electrophoresis through a packed adsorbent column are

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made by LKB-Produkter AB, Sweden (U.S. Distributor: LKB Instruments, Inc., Washington 14, D. C.). The Column Electrophoresis Apparatus (Model 3340C with 1200 v, 60 ma power supply, $725) consists of a 280-ml column tube, which communicates with anode and cathode vessels containing platinum electrodes, and the tubing, vessels, and fittings necessary for the charging of the column with sample, development of a pattern, and elution and collection of the separate fractions. The separation capacity is 10–500 mg of proteins per run, and similar quantities of other types of specimens.

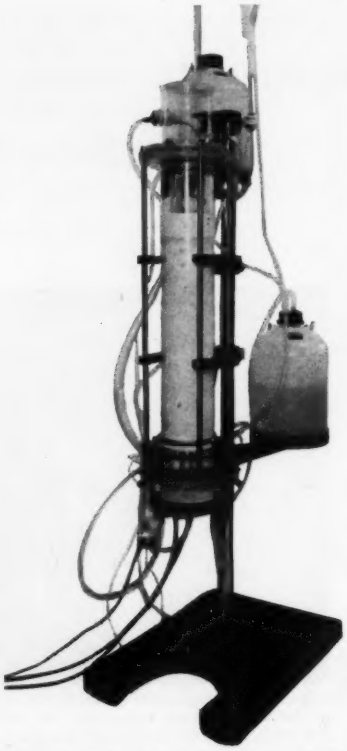
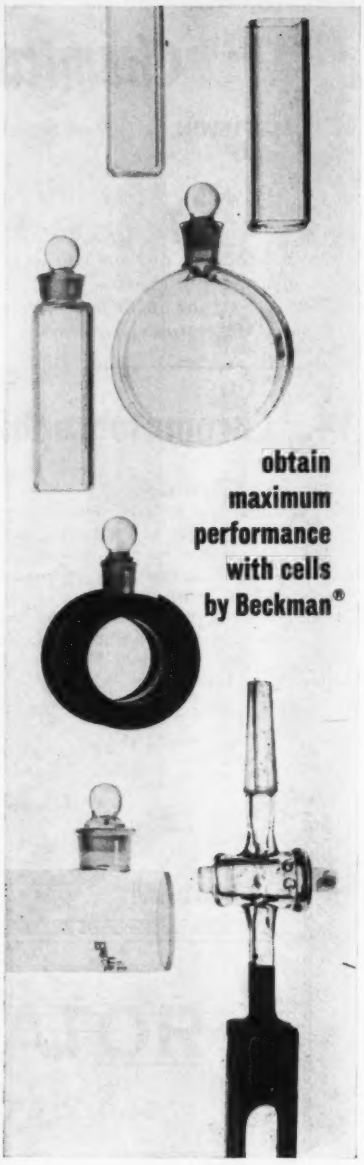


Figure 50. Preparative-scale column electrophoresis apparatus made by LKB-Produkter AB.

For preparative scale work, this manufacturer has available the Porath Electrophoresis Column, Size 60 (Model 5800 A) shown in Figure 50. The column is composed of four concentric polymethacrylate tubes; as much as 30–200 ml of sample may be placed on the column, corresponding to up to 20 grams of dry substance. The column is cooled by means of circulating cold tap water. Voltage is applied to platinum electrodes situated in compartments at the top and bottom of the column respectively. A pump circulates buffer solution continuously through the electrode compartments from a 10-liter reservoir.

The separated components are eluted from the bottom of the column. Fast moving components reaching the out-flow tube are collected continuously during electrophoresis; the slower components are displaced from the column by a suitable buffer flow after the potential has

(*Continued on page A794*)



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GEL ELECTROPHORESIS

Electrophoresis on gel supporting media affords much improved resolution in comparison with paper-strip or free-solution electrophoresis. As many as 24 distinctly-separated components can be obtained from serum samples. Altho equally suitable for micro quantities, gel electrophoresis also accepts much larger quantities making it a practical method for preparative separations on a semi-micro scale; up to 200 mg. of protein mixture can be separated on one gel slab. If direct-contact cooling of the gel slab is employed, complete serum protein patterns can be obtained in less than two hours, showing better resolution than a 20-hour pattern on paper electrophoresis.

The gel material used as supporting medium may be hydrolyzed starch, agar, or the synthetic polyacrylamide Cyanogum (Reg. American Cyanamid Co.). Each of these media gives a different type of resolution, and in many problems it will be advantageous to employ all three concurrently or successively; the procedure then becomes analogous to two-dimensional chromatography. Migration thru a gel slab in the horizontal position introduces undesirable gravitational-convection effects in the migrating zone; therefore, a vertical gel slab is preferable, providing that a proper supporting apparatus is available.

COUNTERCURRENT DISTRIBUTION

Counter-current extraction is a method of analyzing or separating the components of a mixture by dissolving it in one phase of a two-phase solvent system, and extracting it with the other phase. This effects a relative separation of the components according to the distribution coefficients of each. Repeating the extraction many times in a systemic manner multiplies the effects of even small differences in distribution coefficients, so that closely similar substances can be separated. The separated volumes may be analyzed by any applicable method, preferably one which gives the total quantity of solute in each volume. Thus the total volume of solvent may be titrated when dealing with a mixture of acids. The most generally applicable, when non-volatile solutes are concerned, is the determination of the weight of solute in each fraction. It should be noted, however, that macro quantities of material are not required. Extremely low concentrations may be used, which may be far below those required for weight determinations. Necessary, of course, are sensitive analytical methods, such as color reactions or ultraviolet absorption. With such methods, counter-current extraction can analyze quantities as small as those in any other analysis. Results using low concentrations may be even more accurate than those with high concentrations owing to a closer approach to the laws of ideal solutions from which the following equations are derived. Counter-current extraction follows very exactly the Distribution Law

$$Ki = \frac{(i)\,1}{(i)\,2}$$

where Ki is a constant characteristic of the compound i; $(i)_1$, and $(i)_2$ are the concentrations of compound i at equilibrium in phase 1 and phase 2 of the solvent system. This fact allows a very mathematical analysis of the quantitative results obtained in counter-current extraction, and permits the direct comparison of experimental and theoretical results.

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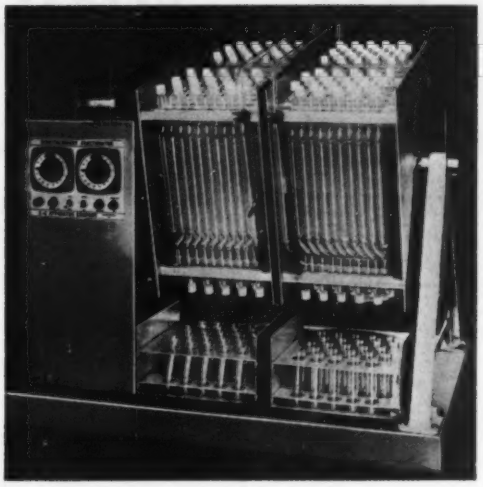

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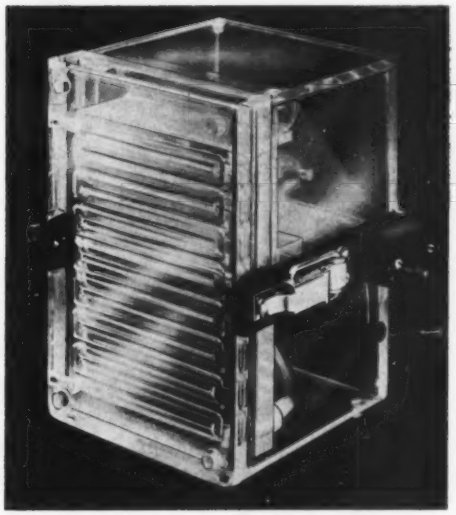

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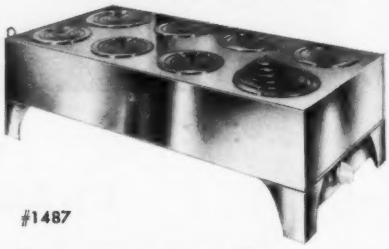

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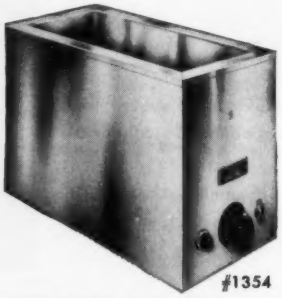

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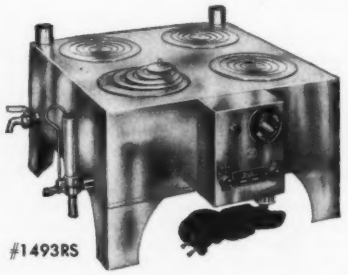

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SQUARE BATHS

Eight models are constructed of same materials as the rectangular types. Four are electric models made with or without thermostatic control. These are also furnished with drain-cock, water level, concentric rings and tubulations. Except for gas-heated models, baths are integrally mounted on attractive supports of the same metal.

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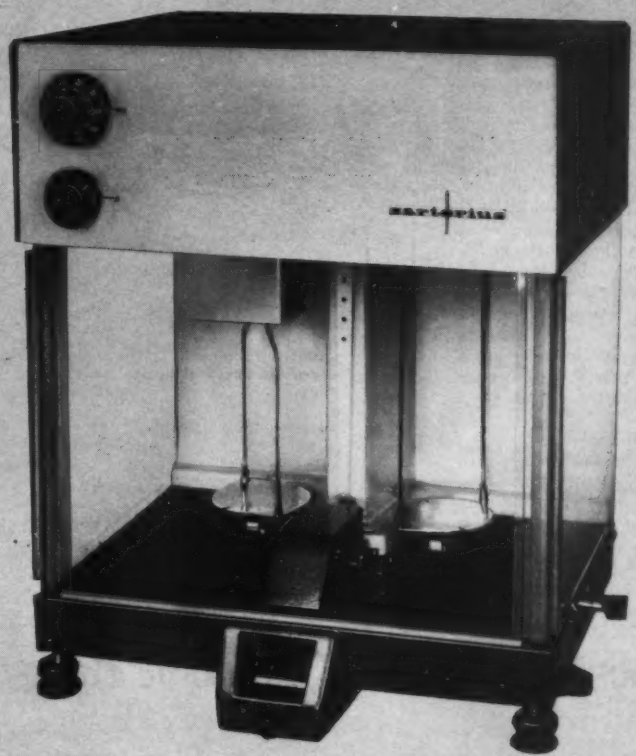

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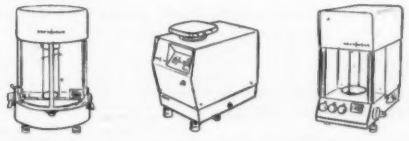

A demonstration or trial of any Sartorius balance may be arranged through your nearest Sartorius distributor. For further information on Series 1500 balances, contact:

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been removed. The column is 60 cm (about 2 feet) long, and has a volume of about 4 liters. Approximately 1–2 liters/min of cooling water is used.

Other Electrophoresis Units

A conventional apparatus capable of accepting up to 12 one-inch wide paper strips is available from Labline, Inc., Chicago 22, Illinois (Electrograf, complete with 500 v, 50 ma power supply, $400). The over-all size of the migration chamber is 15″ × 11″ × 4″, and it is designed so that several units may be stacked for multiple operation, as illustrated in Figure 51. The power supply has four sets of output plugs so that four chambers can conveniently be attached in parallel, as in the Figure. The cover slides onto the body of the chamber in the horizontal plane; if the cover is not completely closed it is not possible to attach the power leads to the chamber electrodes. Hence access to the interior is permitted only when the power is off. This is a useful safety feature; other instruments achieve a similar end by incorporating a microswitch upon which the top cover rests and which breaks the circuit when the cover is lifted.

The Bird Electrophoresis Apparatus ($275, with 300 v, 50 ma power supply) is available from Phipps and Bird, Inc., Richmond, Virginia. It is a conventional instrument, with paper strips supported on two end rods of 1/2 inch plastic rod 19.5 cm apart, and a center bar to give a slight ridge-pole effect.

An electrophoresis instrument for use with paper strips or with gel beds is manufactured by Arthur H. Thomas Co., Philadelphia 5, Pennsylvania (No. 4937-W5, with 300 v, 50 ma power supply, $345). The carrier for paper strips or sheet has an open area of 7 1/4 × 7 1/4 inches. This instrument, introduced about 1953, was one of the first commercially available paper electrophoresis devices.

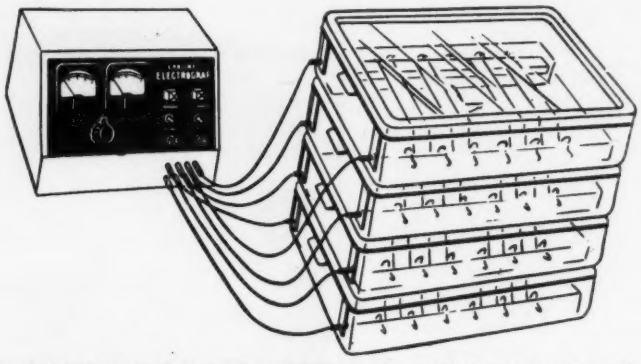


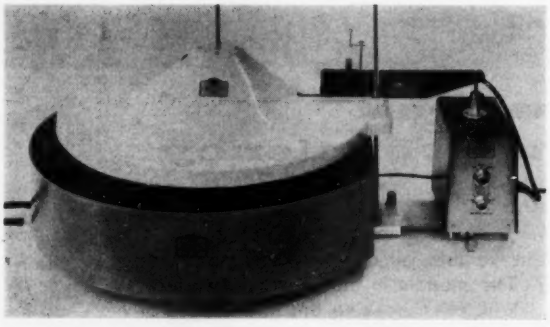
Figure 51. Labline Electrograf, showing four migration chambers stacked on each other, and operated from the four parallel outputs of the power supply.

Thin-Layer Chromatographs

A modification of column adsorption chromatography that has proved to be extremely versatile and successful since its popularization by Stahl and others in 1960 (almost ten years after its initial description by Kirchner, Miller, and Keller) is the technique now known as *thin-layer chromatography, TLC*. It is essentially column chromatography without the column. Or, it is paper chromatography with a column-type adsorbent in place of the paper. That is, a thin, uniform layer of the adsorbent (e.g., silica or alumina) is spread on a flat glass plate, and this sheet of material is then employed in much the same manner as a paper sheet is in ascending chromatography. In order to make the adsorbent hold together as a thin sheet, and adhere to the glass plate, it is mixed with a binder, $CaSO_4$

(*Continued on page A798*)

NEW COOLING TROUGH for LKB AUTOMATIC RadiRac FRACTION COLLECTOR



Where coldroom space is a problem and fraction collecting must be carried out, it is now possible to employ a new accessory from LKB that will allow test tubes to be kept at cold temperatures even though the entire apparatus is used outside of the coldroom.

Standard cold baths with circulating pumps can be employed to expose the Fraction Collector test tubes to cold temperatures by connection to the LKB 3455A Cooling Trough.

The LKB Drop Counter is shown positioned in one of the many operational possibilities for the LKB RadiRac Fraction Collector. The new LKB 3455A Cooling Trough and Dust Cover Assembly are also pictured.

For complete versatility in fraction collection, consider the following LKB features:

- New 3455A Cooling Trough
- Collection in 240 test tubes either by LKB pioneered RadiRac method or conventional circular and spiral methods
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- Large volume assembly easily attachable
- Drop counting, Volumetric, or Time modes of collection
- Siphons for 1 ml, 2 ml, 5 ml, 10 ml, 25 ml, 50 ml
- External marking plug available for easy connection to Recorders in conjunction with automatic analysis of streaming fluids

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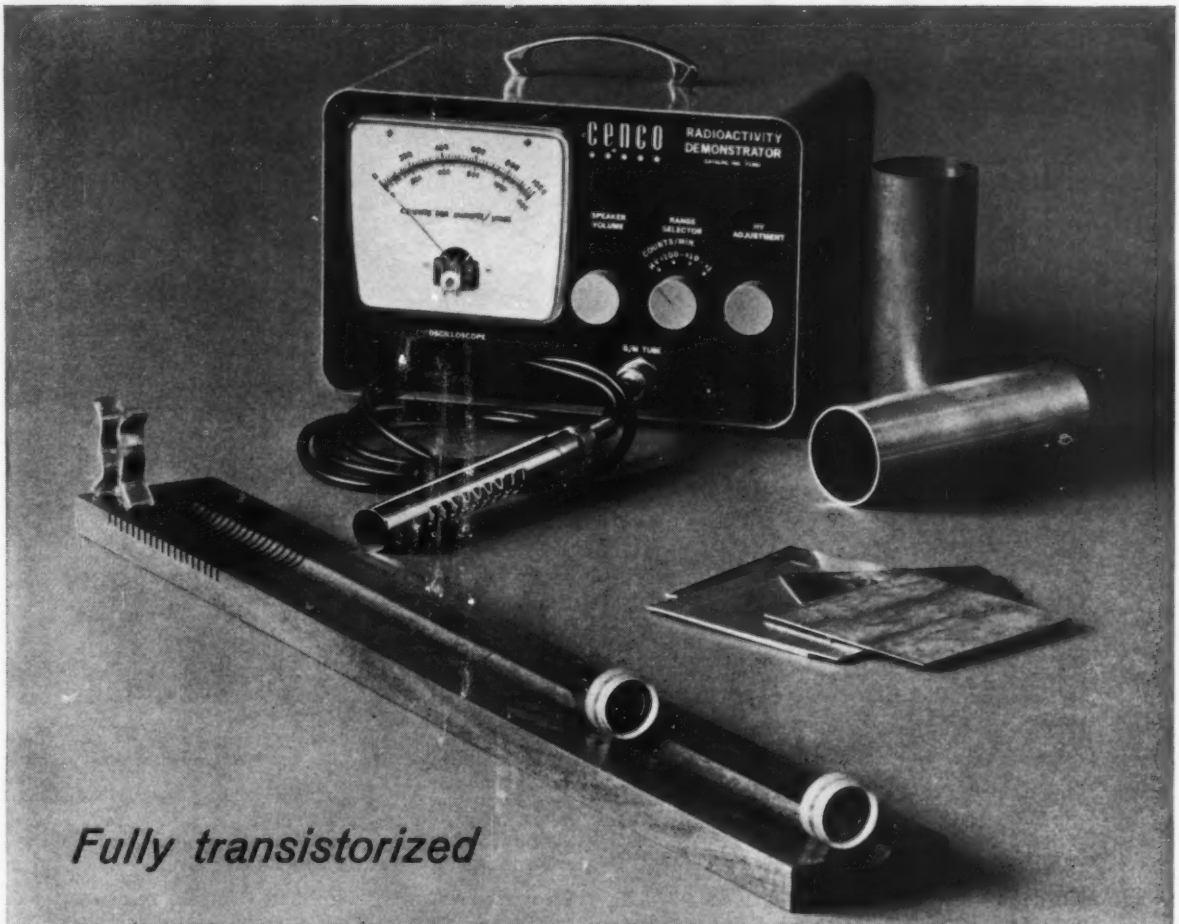
International Sales Headquarters: LKB-Produkter AB, P.O.Box 12220, Stockholm 12, Sweden.



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Rugged, lightweight, long-life transistors requiring no warm-up, are the features of Cenco's new Radioactivity Demonstrator to teach the fundamentals of nuclear radiation to high school and college classes.

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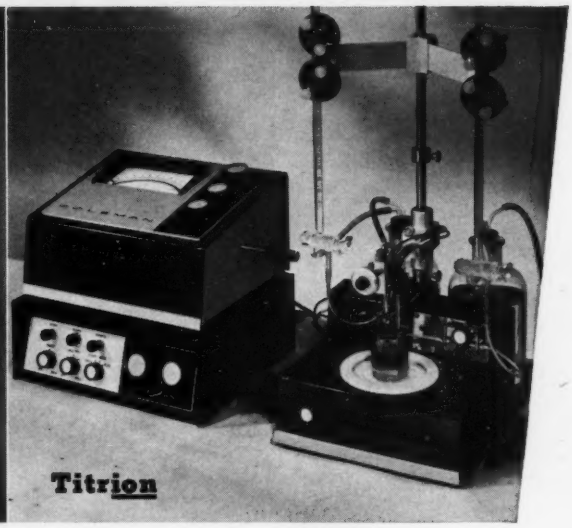

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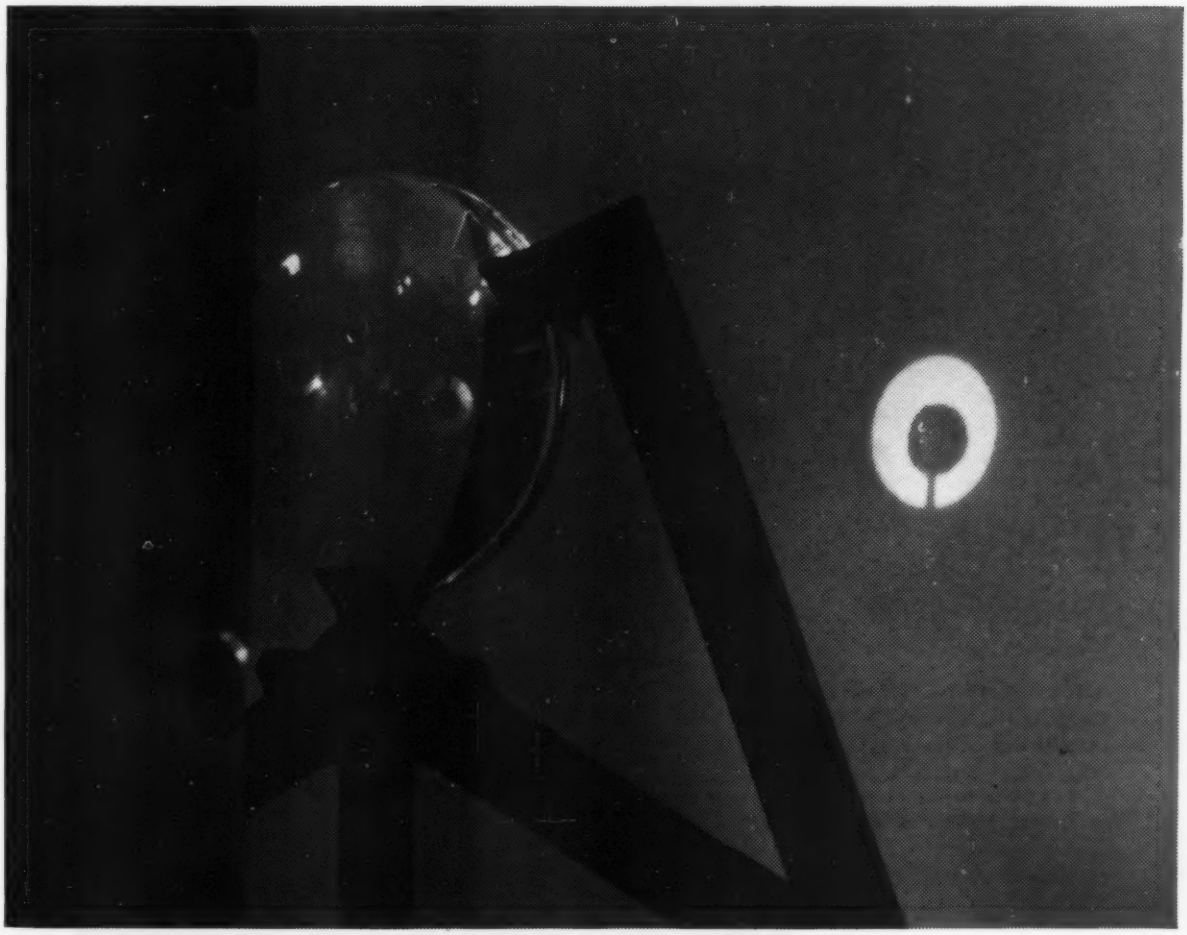
from abstract ideas...fundamental knowledge at Esso Research



Adsorption rather than diffusion may be the controlling mechanism in the transport of liquids through elastomers.

This new hypothesis is supported by evidence obtained by Esso Research scientists using a novel photo shadow method for continuous measurement of swell in elastomers. As a result of these fundamental studies, more intimate knowledge of structure and increased understanding of segmental motion may make possible more precise predictions of polymer behavior.

Increasing the cross-link density in an elastomer network appears to decrease the diffusion coefficient to an extent not predicted by the diffusion theory. Observations also show the seeming contradiction that increasing the path length with immobile obstructions, in the form of a variety of carbon black particles, also increases the apparent diffusion rate 2.5 times.

Re-examination of well known equilibrium uptake data poses such questions as: Why does a butyl rubber vulcanizate sorb twice as much cyclohexane as n-hexane when the latter has a much more flexible structure? How does a butyl elastomer distinguish between the flat plate structure of benzene and the chair structure of cyclohexane whereas natural rubber does not?

Critical experimentation, together with mathematical studies, have strengthened the thesis that the solvent molecules pass into the elastomer by successive adsorption on successive planes. The elastomer chains appear to play an active role in the transport process rather than the passive one required by diffusion.

...adapted from a scientist's notes at Esso Research and Engineering Company P. O. Box 45B, Linden, New Jersey.

(i.e., plaster of paris, which hydrates when treated with water to form gypsum). The thinness of the layer of adsorbent coupled with the adsorption and fluid flow characteristics of the solid, result in very rapid migration of constituents during chromatographic development. Thus, in TLC a complete run, in which the components of a specimen may migrate 10–15 cm, is generally completed in 20–40 minutes. The fact that the adsorbent is exposed is also very useful, for the location of the components can be readily determined by spray reagents, and many reagents which cannot be used on paper are suitable for use with the inorganic adsorbents involved in TLC.

The essence of the instrumentation required for TLC is a means of producing the thin layer of the adsorbent-plaster slurry. The first commercial device for this purpose was that made by C. Desaga, Heidelberg, Germany (U. S. distributor, C. A. Brinkmann and Co., Great Neck, N. Y.). The applicator made by this supplier is shown in Figure 52. It consists of a reservoir for the slurry, and a channel between the underside of the trailing edge of the applicator and the plate beneath it. The operation of the applicator is shown in schematic diagram in Figure 53. The applicator shown in Figure 52 leaves a layer with a fixed thickness of 0.275 mm. (No. 250015, $176). Another model is available in which a screw adjustment permits choice

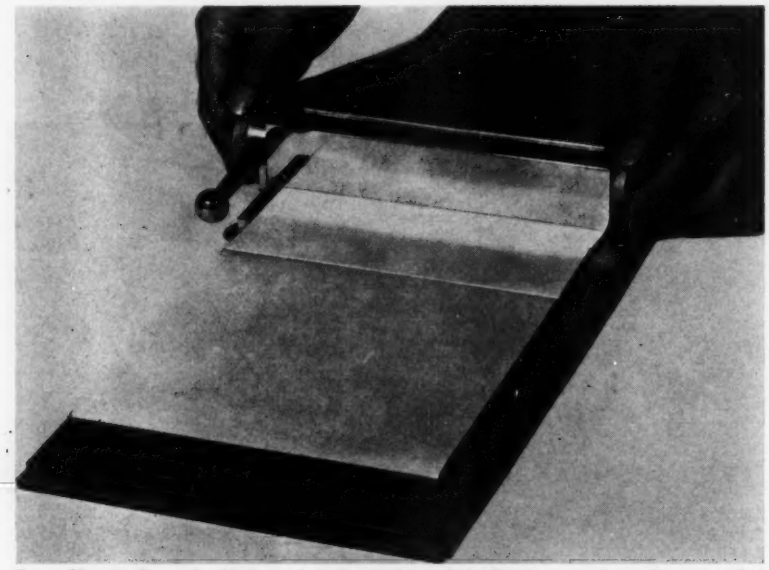


Figure 52. Applicator for thin-layer chromatography made by Desaga. As the applicator is pulled along, a thin, uniform layer of the white slurry is left on the glass plate.

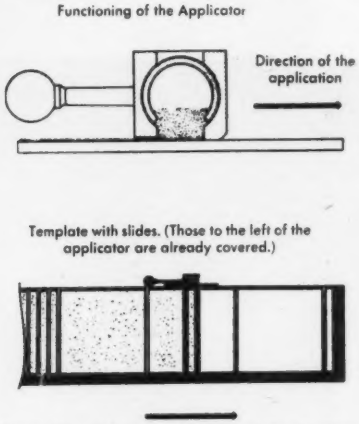

Functioning of the Applicator
Direction of the application
Template with slides. (Those to the left of the applicator are already covered.)

Figure 53. Schematic diagram of the functioning of the thin-layer applicator.

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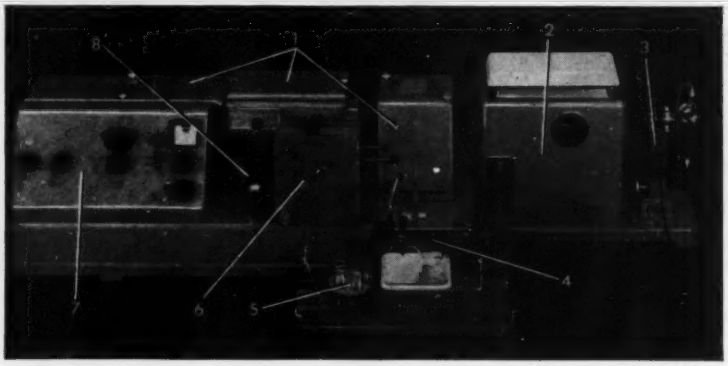

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of the layer thickness up to 2.0 mm. (No. 250010, $215). A kit for TLC, including an applicator, a template for positioning the specimens on a plate, and appropriate glassware and handling accessories is also available (No. 250000, with non-adjustable applicator, $332).

TLC equipment is also manufactured by Research Specialties Co., Richmond, California. The applicator used, shown

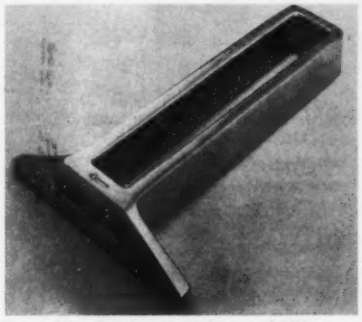

Figure 54. Thin-layer applicator of RSCo.

in Figure 54, is simpler in design than that described above. It produces a layer 0.25 mm thick (Model 200, $45). A kit, containing the applicator and other useful handling equipment, including adsorbents, spray reagents and pipets, is available (No. A-200, $260).

Photometric Effluent Sensing Systems

A variety of instrumentation is available for the continuous, automatic sensing of the composition of the effluent issuing from a chromatographic column. A simple, absorptiometric approach to this problem is exemplified in the Uvicord Ultraviolet Absorptiometer of LKB-Produkter AB (No. 4701A, $720). The design of the sensing unit of this instrument is indicated schematically in Figure 55.

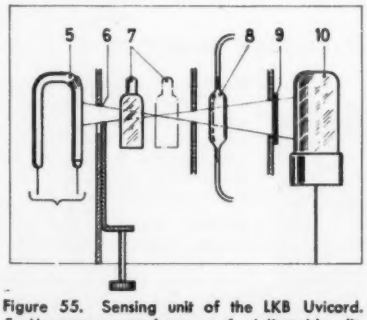

Figure 55. Sensing unit of the LKB Uvicord. 5, Mercury vapor lamp. 6, Adjustable slit. 7, Liquid filter(s); $NiSO_4$ solution passes 2537 A, but removes all but 8–10% of the 3126, 3132 A lines; CS_2 may be used in a second filter to remove these. 8, Flow cuvette. 9, Black glass filter to remove any stray or fluorescent light. 10, Phototube.

Light from a mercury vapor lamp is filtered to isolate the 2537 A line. This passes through a flow cuvette, through which the effluent from the column is flowing, and then enters a phototube. As various components which absorb ultraviolet radiation emerge from the column, they reduce the intensity of the

(*Continued on page A807*)

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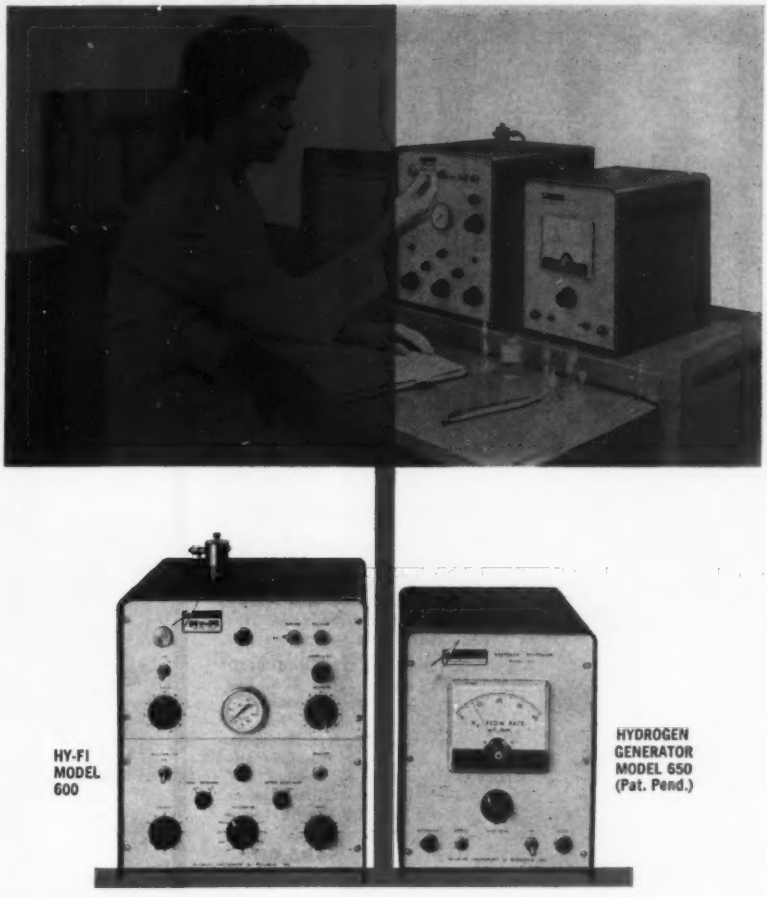

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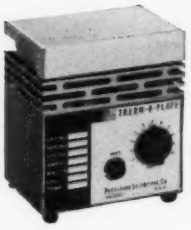

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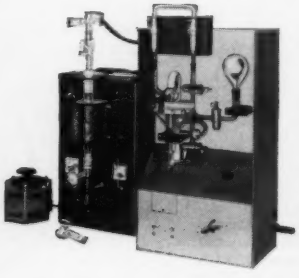
(Similar models are also available in 200 and 500 gram capacity.)

LECO HYDROGEN ANALYZER



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transistorized nuclear instruments

The new Designer Series is the most complete group of instruments ever presented to the nuclear field. Unlimited possibilities, of combining instruments to design special systems, are possible by the simple arrangement or rearrangement of the plug-in instruments.

The first six of this extensive line of transistorized small instruments, now available, greatly improve instrumentation versatility for both small and large educational laboratories and research facilities.

By plugging-in the basic instruments needed to form a system, the user easily prepares for an experiment in minimum set-up time and is assured of perfect system interconnections.

The basic unit common to all systems is a compact instrument case with a self-contained power supply and an internally wired power and control distribution system, the Model 29-1. Instrument Case and Power Supply has built-in compartments which allow the user to position and plug-in four single instruments, or two double instruments (each occupying two compartment positions), or any combination of these. The Model 29-1 Instrument Case weighs 35 pounds and is 19" wide x 8¾" high and 15½" deep.

Development is continuing on many additional plug-in instruments to complement the six now available. The introduction of each new instrument by RIDL will greatly expand overall system capabilities of the Designer Series. Thus, laboratories may purchase instrument packages to meet both current and changing experimental requirements.

Other functional instruments now in final stages of development are: additional High Voltage Power Supplies and Amplifiers, Single-Channel Pulse Height Analyzer and Electronic Sweep, additional Scalers, Scamp System, Rate Meters, and Electrometer.

Side view of Designer Series Scaler.

MODEL 54-7
FIVE DECADE ELECTRONIC TIMER

The Model 54-7 is an electronic timer using the 60 cycle AC line frequency as the primary time base; providing two time ranges with increments of 1/10 minutes and 1/10 seconds.

The time scaler operates as a slave device, depending on external commands for start, stop and reset operations.

SPECIFICATIONS

Ranges:.....10^3 minutes, 0.1 minute increments.
10^3 seconds, 0.1 second increments.
Time Base:.....60 cycles from A.C. line.

MODEL 2-9
2-Pi GAS FLOW COUNTER

The heart of the Model 2-9 Gas Flow Counter is a specially designed 2-Pi geometry gas flow counter incorporating a convenient sliding drawer sample holder. The self-contained circuits include a (1) proportional pre-amplifier, (2) gain-of-400 linear amplifier, and (3) discriminator which provides four switch-selected discrimination levels (1, 10, 20, and 300mv).

The entire gas flow control system, with the exception of the gas supply, is also contained within the instrument itself. Front panel PURGE and BUBBLE rate valves, and the visual bubble rate indicator, allow flow rate to be adjusted and continuously monitored.

An automatic gas purge system interlocks with the sample drawer lock and initiates a 30 second gas purge each time the drawer LOCK is opened and closed.

The amplifier and discriminator sections are identical to the Model 30-19 Amplifier and Discriminator instrument.

SPECIFICATIONS

Counter Geometry:.....2-Pi.
Sample Size:.....Opening is 7/16" deep and 2½" diameter.
Gas Fill:.....Geiger Region: Helium Isobutane
Proportional Region: Methane or P-10.
Discriminator Output:...+10 volts; 1 μ sec wide.
Background:.....α-0.1 cpm
β & γ 60 cpm

MODEL 49-26
SIX DECADE PRESET COUNT SCALER

The model 49-26 instrument is a 1 microsecond, six decade preset count scaler. Preset is accomplished by means of six 10-position PRESET COUNT thumb wheel switches mounted as a part of each decade. These switches may be set to monitor any one of the 10 decoded outputs on each decade and present a signal to the preset circuitry when the preset state is reached.

SPECIFICATIONS

Input:	10 volt positive pulse.
Count Capacity:	10^6.
Resolution:	Approximately 1 μ sec.
Preset Count Selection:	0 to 1,000,000 all inclusive.
Input Impedance:	220 mmf capacitor in series with approximately 1000 ohms.
Maximum Continuous Count Rate:	150 KC with 6 decade preset 1 MC with 5 decade preset.

MODEL 40-8
HIGH VOLTAGE POWER SUPPLY

The Model 40-8 High Voltage Power Supply employs a full wave voltage tripler, which is applied across a string of sixteen 85A2 reference tubes. The high voltage power supply is primarily used with scintillation counters where load impedance does not change widely.

SPECIFICATIONS

Range:	595—1350 volts D.C.
Polarity:	Positive.
Control:	10 position switch with 85 volt increments.
Regulation:	0.005% variation per one volt change in line voltage.
Current:	1 milliampere.
Output impedance:	100,000 ohms.
Ripple:	Less than 50 millivolts.
Stability:	0.01% per degree centigrade.

MODEL 30-19
AMPLIFIER AND DISCRIMINATOR

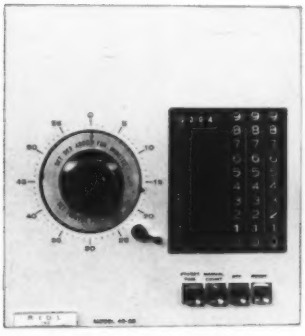
The RIDL Model 30-19 Amplifier and Discriminator design is based on that used in RIDL Multichannel Analyzers. The amplifier output is linearly related to the input; the discriminator output is a fixed amplitude generated whenever an output from the amplifier exceeds the adjustable triggering level.

The amplifier section has two cascaded gain-of-20 feedback loops providing a gain of 400 and driving a complementary emitter-follower output stage.

The discriminator provides an output of constant width and amplitude whenever an input pulse exceeds the preset triggering level. The triggering level may be varied continuously over the amplifier's dynamic range.

AMPLIFIER	SPECIFICATIONS
Input Impedance:	1,000 to 32,000 ohms dependent on amplifier gain setting.
Input Pulse Polarity:	Positive.
Maximum Gain:	400.
Control Range:	64:1.
Rise Time:	Approximately 0.25 μ sec.
Fall Time:	Same as input signal.
Output:	Positive 0-10 volts.

DISCRIMINATOR	
Range:	0.1-10 volts—controlled with 10-turn Helipot. 1, 3, 10, 30, 100, 300 millivolt fixed sensitivity setting.
Input Polarity:	Positive—direct from amplifier output.
Resolution:	Approximately 1 μ sec.
Linearity:	0.25%.
Output:	10 volts positive.



MODEL 49-25
SEVEN DECADE ELECTRO-MECHANICAL SCALER-TIMER

Basically, the Model 49-25 is a combined scaler and timer. As a double instrument package, it occupies two positions when inserted in the Instrument Case. The scaling portion is composed of three cascaded 1 microsecond decades driving a 4-digit electrically reset mechanical register. The combination permits total capacity of 10^7 counts.

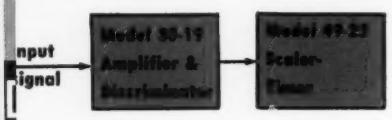
The mechanical timer may be selectively preset at 1 second intervals anywhere between 0 and the maximum time capacity of 60 minutes. A momentary-type RESET button resets the decades and register.

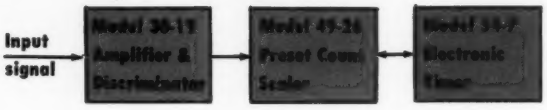
SCALER	SPECIFICATIONS
Input:	10 volt positive pulse.
Resolution:	Approximately 1 μ sec.
Mechanical:	4 digit, electrically reset maximum—20 pps.
Capacity:	3 decades electronic, 4 decades mechanical, total 10^7.
Maximum Continuous Count Rate:	Approximately 20,000 pps—rate limited by mechanical register.

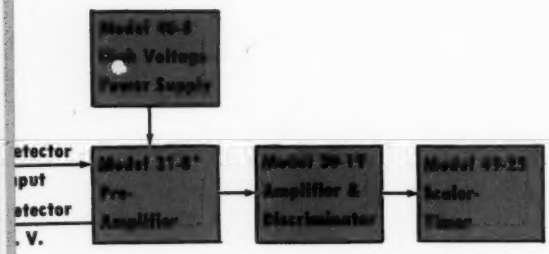
TIMER	
Preset Time Range:	1 second to 1 hour in 1 second intervals. Accurate to 0.1 seconds.

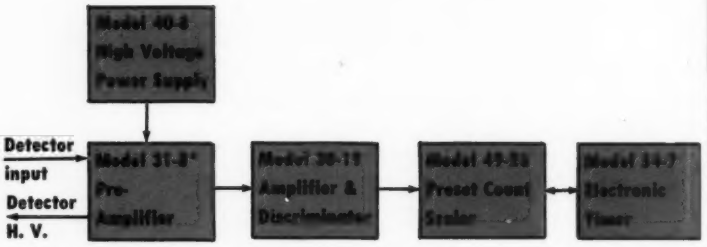
RIDL DESIGNER SERIES SYSTEMS

Following are several systems which you can design from the RIDL Designer Series units now available.



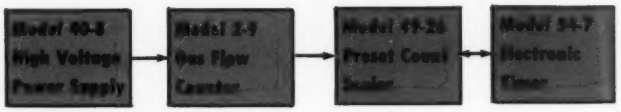






*The Model 31-8 is a standard RIDL Transistorized Preamplifier which may be used with detectors operating at voltages up to 2000 V. Conversion gain is approximately one volt per 5 X 10-11 coulombs. Dimensions are 2⅞" x 3⅛".

All systems include the Model 29-1 Case and Power Supply, and all necessary interconnecting cables.



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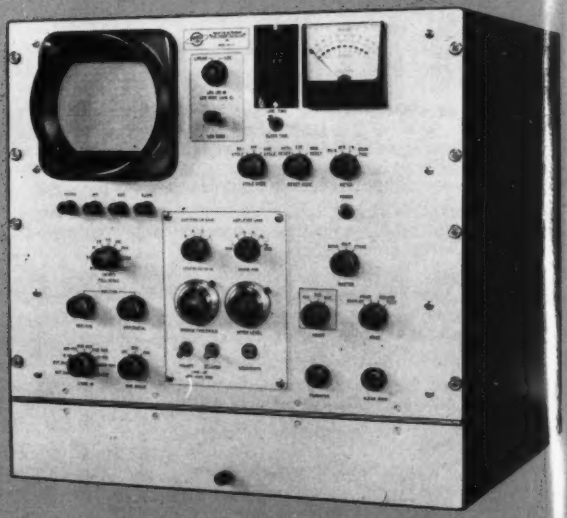

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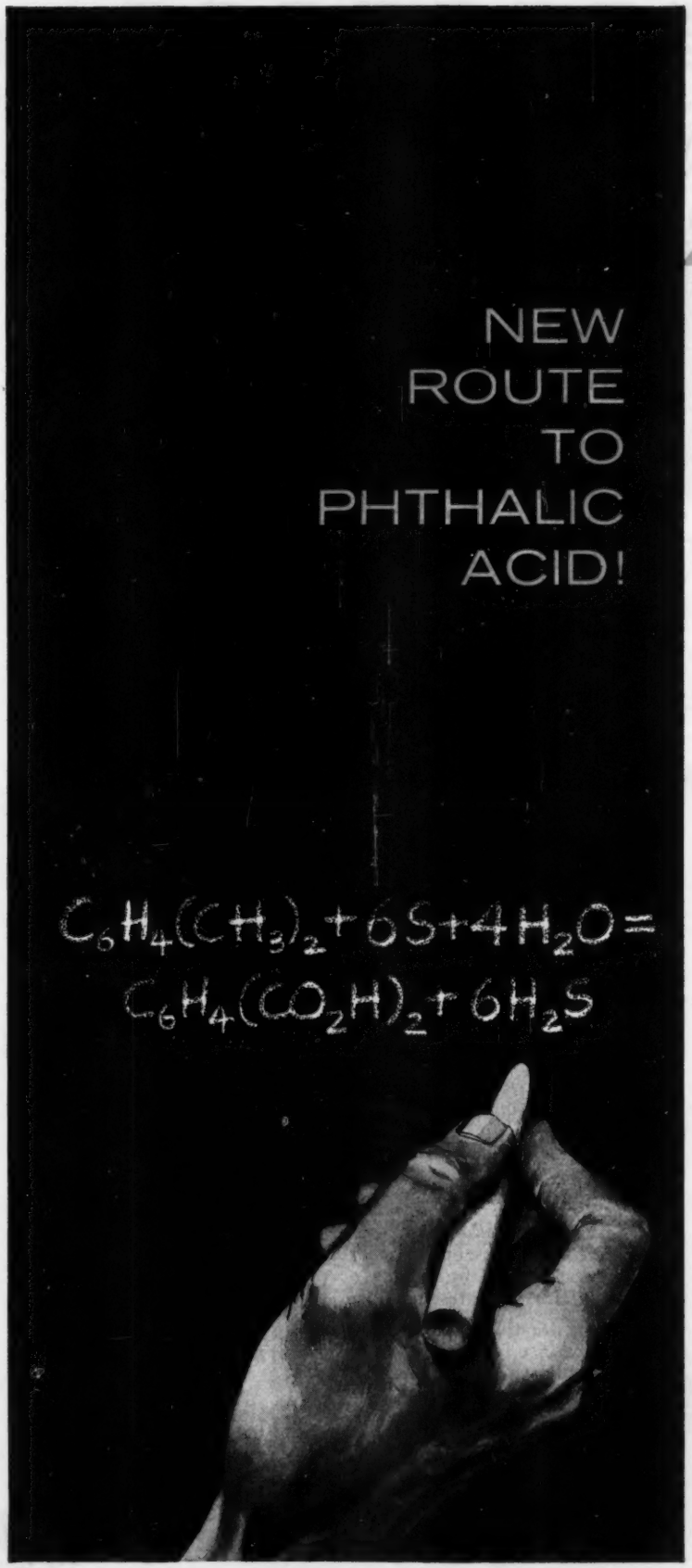

SULPHUR
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In a report to the Petroleum Division of the ACS, Dr. William G. Toland of the California Research Corporation recently announced the development of a new method for producing Phthalic Acid, with the following over-all reaction:

$$C_6H_4(CH_3)_2 + 6S + 4H_2O = C_6H_4(CO_2H)_2 + 6H_2S$$

Note that the presence of Sulphur right smack in the middle of this reaction is necessary for its completion. The Sulphur leaves the process at this point. The hydrogen sulphide can be oxidized to elemental Sulphur and returned to the initial reaction. It is thus operable in a closed cycle.

According to Dr. Toland, who developed this new route to phthalic acid, the process is simple and capable of high recoveries.

Here is another good example of how strongly Sulphur is in the processing picture. Together with its many derivatives it enters into countless reactions.

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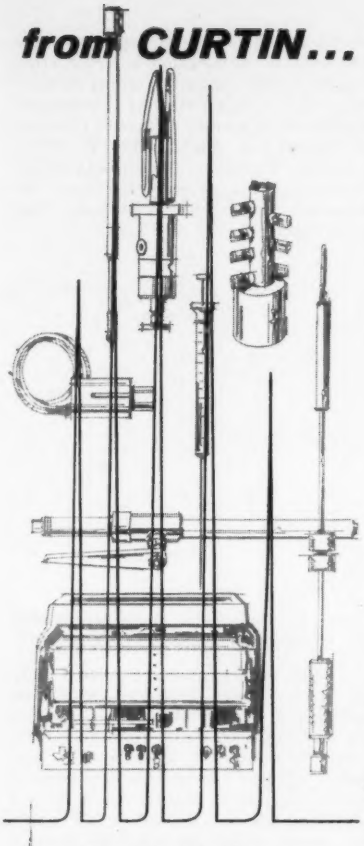

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141

light impinging on the phototube, and the changes in the output of the latter signals their presence in the effluent. Several cells may be employed as the flow cuvettes, with optical paths ranging from 1 to 5 mm, corresponding to cell volumes between 0.05 and 0.3 ml. Using a 5 mm cell, a 10 mm pen deflection of a recorder attached to the output of the Uvicord is obtained with 0.02% serum albumin in 1% NaCl, or with 0.00036% tyrosine in 0.1 *N* NaOH.

LKB also produces a conductance sensing device, which monitors the conductivity of the effluent as it flows through a cuvette fitted with platinum electrodes (Conductolyzer, $890). On the range 0–500 ohms, a change of 10 ohms will produce a full-scale deflection of the recorder; on the 0–10,000 ohm range, a variation of ±50 ohms produces the same effect.

An ultraviolet absorptiometer sensing unit similar in design to the LKB instrument described above is manufactured by Buchler Instruments, Inc., New York 31, N. Y. (Uviscan, $570). A photograph of this unit attached to a chromatographic column and monitoring the effluent before it falls into a tube in a fraction collector is shown in Figure 56.

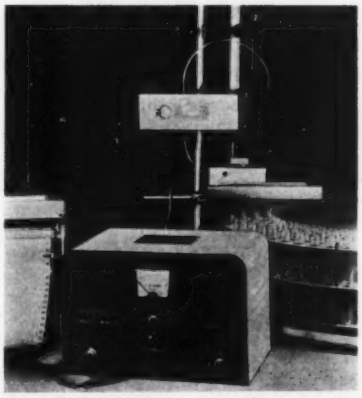


Figure 56. Buchler Instruments Uviscan sensing unit (middle of figure) and amplifier control unit (lower foreground) shown attached to a chromatographic column. Effluent from the column flows through the sensor unit and then drips into a tube mounted in the fraction collector.

A three-channel recording flow absorptiometer, shown in schematic outline in Figure 57, is made by Research Specialties Co., Richmond, California (Recording Flow Photometer, $3870). The three photometer channels operate with a single absorption cell through which the sample flows. On the 3-point recorder, the optical density in each of the channels is recorded by means of red, green, and blue dots. Measurement of sample flow is accomplished with a bubble flow meter. Channel 1 measures at a wavelength of 440 millimicrons; channels 2 and 3 at 570 mμ. Channel 3 has an optical path length one-third that of channel 2, so that a wide range of optical densities can be covered.

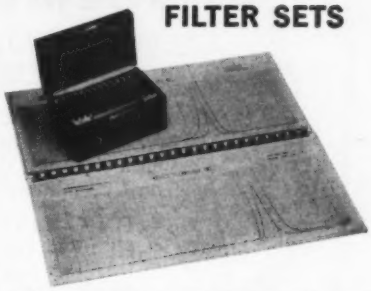
This type of three-channel recording photometer is very useful in the automated amino acid analysis technique developed by Spackman, Stein, and Moore, and

(*Continued on page A808*)

WAVELENGTH REJECTION FROM X-BAND TO X-RAY

MONO PASS

INTERFERENCE FILTER SETS



Optics Technology offers filter sets covering the range of 400 millimicrons to 2.75 microns to meet your requirements in spectral analysis. These MONOPASS interference filter designs reject from X-band to X-ray! The possibility of spurious results is eliminated as only the desired wavelength is passed. Calibration curves for each filter pinpoint accuracy. The curves for each set are laminated in plastic and bound in a rugged volume.

Visible Spectrum Set 10A includes ten MONOPASS filters to isolate principal lines as K, Ca, Hg, etc., from 706 millimicrons to 404 millimicrons, important in flame chemical analysis. Four neutral density filters and a linear spectral "wedge" filter are included. Price, $325.

Visible Spectrum Set 12A includes ten MONOPASS filters uniformly spaced from 400 millimicrons to 700 millimicrons, as well as four neutral density filters and a linear spectral "wedge" filter. Price, $325.00.

Infrared Set 15A includes ten MONOPASS filters spaced at every 0.1 micron between 0.8 micron and 1.75 microns. Price, $450.

Infrared Set 20A includes ten interference filters on 1" diameter substrates spaced at every 0.1 micron between 1.75 microns and 2.75 microns. These filters are blocked out to 3.2 microns on the long end and to X-ray on the short end. Price, $450.00.

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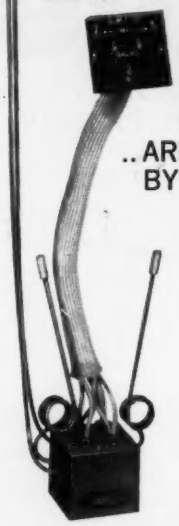

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several manufacturers now produce such instruments. In addition to RSCo, other makers of three-channel recording photometers are: Gilson Medical Electronics, Middleton, Wisconsin; Phoenix Precision Instrument Co., Philadelphia 40, Pennsylvania; Technicon Instruments Corp., Chauncey, New York; Beckman Instruments, Inc., Spinco Division, Palo Alto, California.

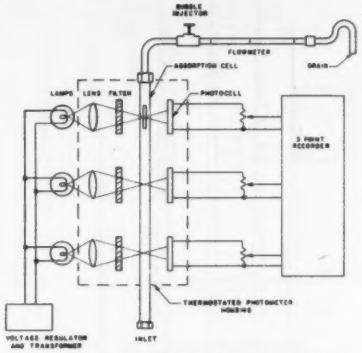


Figure 57. Optical plan of the three-channel flow absorptiometer of Research Specialties Co.

A series of absorptiometers based upon the double-beam, twin-detector optical principle is manufactured for chromatographic applications by Canal Industrial Corp., Bethesda 14, Maryland. The design of these instruments is illustrated by the diagram in Figure 58. A central U.V. light source is flanked on either side by mirrors which direct beams through two quartz cuvettes and interference filters to the photocathodes of two phototubes. The solvent passes through one cuvette, solvent plus eluate passes through the other. The recorder responds to a signal formed by the ratio of the outputs of the phototubes, and hence gives a response that is characteristic of the eluate and independent of any variations that may occur in the absorption properties of the solvent. Several models of these sensing instruments are available, ranging from a fixed wavelength model, through

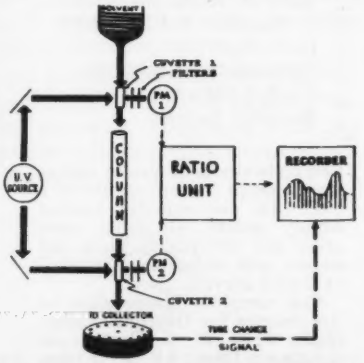
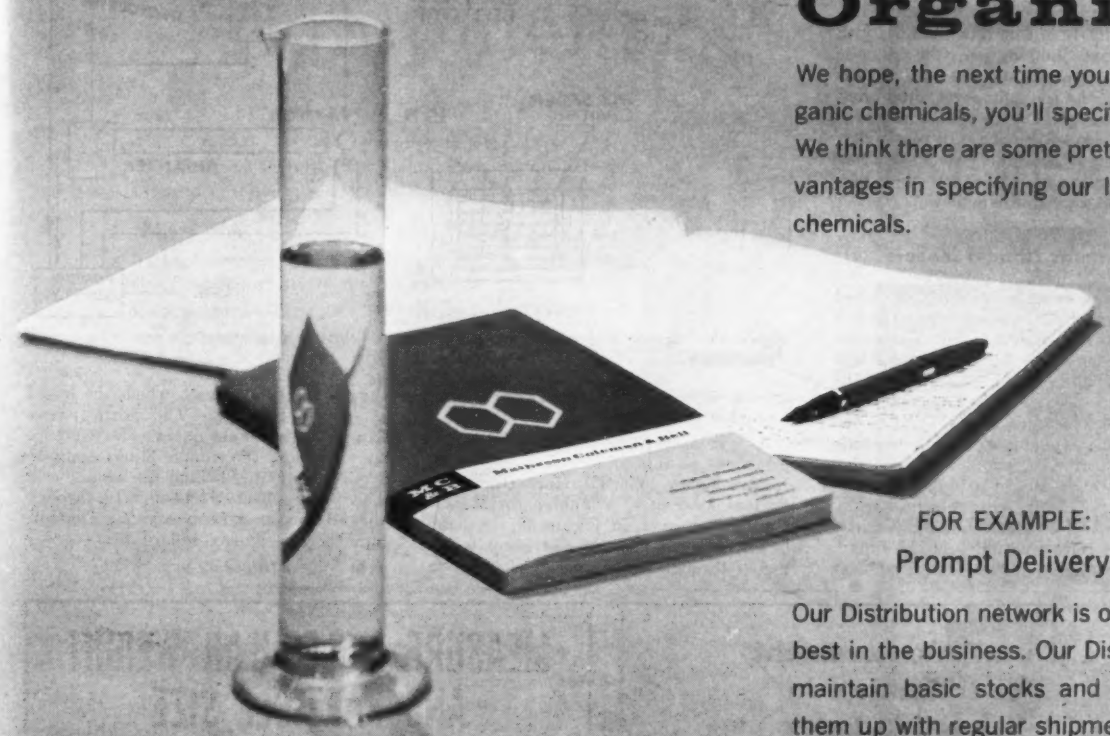


Figure 58. Double-beam, twin-detector design of the flow absorptiometers available from Canal Industrial Corp.

models in which several wavelengths may be selected by the use of interference filters, to a unit with two sensors and two monochromators, permitting a wide range of choice of measuring conditions.

(*Continued on page A810*)



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An instrument that is designed to be attached to a monochromator such as the Beckman Model DU, or the Zeiss Model PMQ 11, and that will record optical densities of the effluent from a chromatographic column at any wavelength in the range of the monochromator, is made by Gilford Instrument Laboratories, Inc., Oberlin, Ohio. The sensing is performed by a 1P28 photomultiplier tube in a special feedback circuit which adjusts the voltage applied to the dynodes of the tube in accordance with the amount of light striking the cathode of this tube. Thus, with very intense illumination, the dynode voltage is decreased and the sensitivity is made low. As the light level diminishes, the voltage applied to the tube is increased with a subsequent increase in sensitivity. Thus, under all conditions the output current is small, and stable, low-noise performance of the sensor is achieved.

Refractometric Effluent Sensors

The photometric sensors described above are selective instruments, and a constituent will be detected in the effluent from a chromatographic column only if it has appreciable absorbance for light of the wavelength used. Many substances of interest are essentially transparent to the wavelengths available in the photometric sensors described above. The refractive index, however, is a relatively non-selective property, in the sense that practically every eluate will contribute significantly to the net refractive index of the liquid. Hence, a number of refractometric effluent sensors have been developed for chromatographic use.

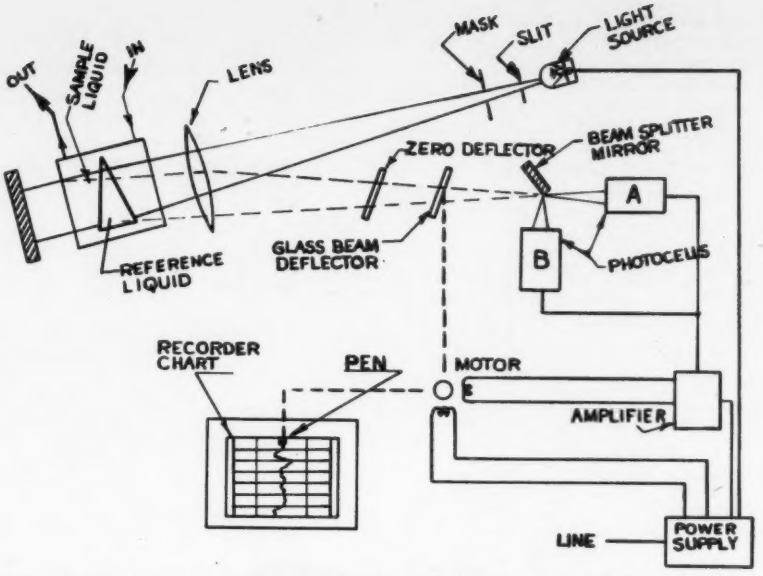
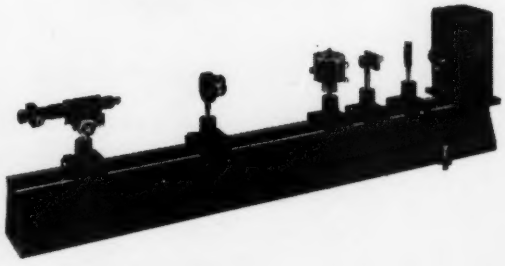


Figure 59. Optical diagram of the design of the Waters Associates Differential Recording Refractometer.

A schematic diagram of the design principle of the Differential Recording Refractometer ($3800) manufactured by Waters Associates, Framingham, Massachusetts, is given in Figure 59. A light beam from an incandescent lamp is directed through a slit, mask, and lens to the sample cell through which the liquid sample is flowing. The beam passes through the sample liquid, the reference standard, and the sample liquid again, to a mirror mounted behind the sample cell. The mirror reflects the beam back through the cell for a second traversal of the sample liquid and reference standard.

(*Continued on page A814*)

Direct Measurement of Difference in Refractive Index



The New Brice-Phoenix Differential Refractometer uses a split cell to measure the difference in refractive index between a dilute solution and its solvent.

Limiting sensitivity 3 x 10^{-6}.

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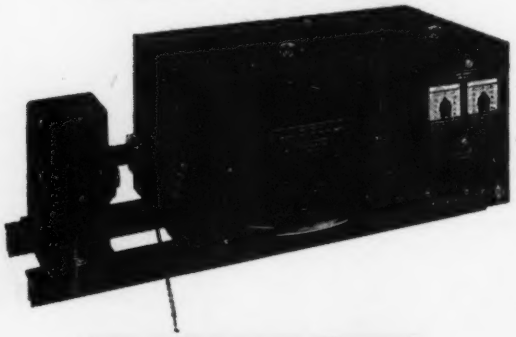
MEASURE MOLECULAR WEIGHT AND PARTICLE SIZE



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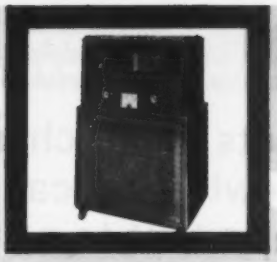
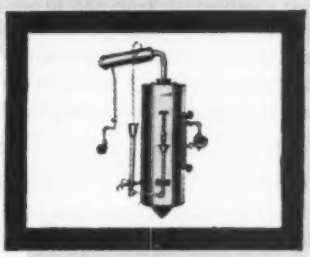
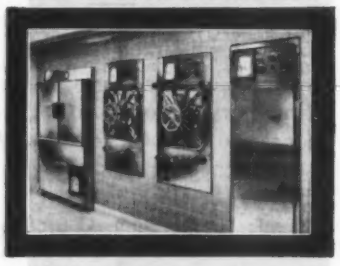
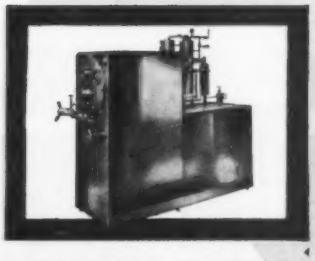
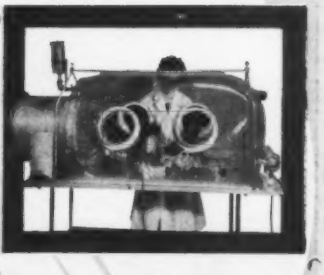
AMSCO Microbiological Equipment











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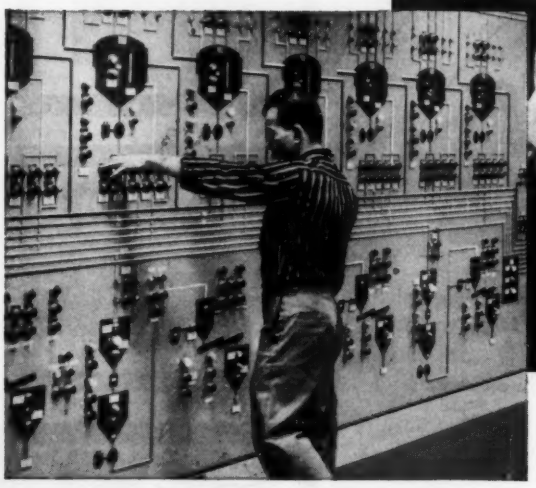

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COMBINING VOLUMES
Hydrogen Chloride and Ammonia

A Chem Ed TESTED DEMONSTRATION

Submitted by: P. A. Seepharaman, The New College, Madras, India
Checked by: Jerry Janssen, Michigan State University, East Lansing

PREPARATION

Obtain a one-liter florence flask, two one-hole rubber stoppers and one solid rubber stopper to fit the flask, a 25-cm length of 7-mm glass tubing, string, a 1-qt capacity polyethylene freezer bag, a pinch clamp, and a short piece of rubber tubing. A source of both hydrogen chloride gas and ammonia gas will be required. Place one of the one-hole rubber stoppers on the glass tube so that it will extend to within 1 cm of the bottom of the flask. Place the second one-hole rubber stopper on the exposed end of the glass tubing with about 2 cm of the tubing extending beyond the stopper. With string, tie the mouth of the plastic bag tightly around the stopper nearest the shorter end of the tubing. Press all of the air out of the bag and fill it with ammonia. When the bag is full, close the end of the tube by placing a short piece of rubber tubing with the pinch clamp on it. Fill the flask with dry HCl gas and close the flask with the solid rubber stopper.

DEMONSTRATION

Remove the rubber tubing and pinch clamp from the glass tube and quickly place the tube in the flask. Press the stopper into the neck of the flask to insure a tight fit. The ammonia gas will slowly diffuse into the flask forming a white cloud of ammonium chloride. The reaction ($NH_3 + HCl$ to give NH_4Cl) will take about five minutes for completion. The walls of the flask become coated with the white ammonium chloride and are hot to the touch. The bag will be deflated at the end of the reaction, showing a marked reduction in volume.

REMARKS

The flask and the plastic bag must be dry. The fittings must be tight. To show the complete change in volume, one is tempted to suggest using polyethylene bags for both reactants. However, the heat of reaction is sufficient to destroy the bag in which the reaction takes place.

This demonstration and another variation using glass cylinders has been described in the *School Science Review*, **42**, 528–529 (1961), John Murray, London.

DEMONSTRATION NOTES

A Chem Ed TESTED DEMONSTRATION

From time to time, items such as the following which do not require a full half-page will be listed together. Some are modifications of previously published demonstrations; they may be clipped and attached to the pertinent experiment.

COMMON ION EFFECT

A neat demonstration of the common ion effect was published in THE JOURNAL, **25,** 489 (1948), by Harvey Sorum of the University of Wisconsin. The demonstration compares the volume of foam and the rate at which it is produced when calcium carbonate is treated with buffered and unbuffered acetic acid solutions. O. A. Taliaferro points out that the desired results are obtained only if precipitated calcium carbonate is used. He suggests Mallinkrodt 4052 light precipitated USP, or Baker precipitated powder USP 1300, or Fred Portz & Son USP light precipitated 2288P.

CHLORINE WATER SUBSTITUTE

Philip Kasimer of Quinnipiac College, Hampton, Connecticut, suggests a solution of chlorine in carbon tetrachloride as a substitute for chlorine water. This solution is stable for over a month and works nicely with the identification of halide ions. See also H. N. Alyea, "Demonstration Abstracts, 20-6s," THIS JOURNAL, **37,** A300 (1960).

RAPID FILTERING WITHOUT USING A FILTER PUMP

Dale E. Rose, Hampton High School, Hampton, Virginia. Obtain a polyethylene bottle (like those sold in stores for dispensing mustard, etc.), squeeze this bottle so as to force out a portion of the air, then insert the narrow tip into the funnel stem. The bottle will then inflate leaving the precipitate on the filter paper and quickly forcing the filtrate into the dispenser.

WRITING ON BEAKERS, FLASKS, ETC.

Dale E. Rose, Hampton High School, Hampton, Virginia, suggests using a "magic marker" purchased from the drugstore for about $.30. This writes quickly on dirty or wet glass, lasts a long time, and is removed simply by rubbing with a cloth dampened with denatured alcohol.

BOILING LIQUID FREEZES WATER

Dale E. Rose, Hampton High School, Hampton, Virginia. Place acetaldehyde in a stoppered side arm flask. Connect the side arm to an aspirator. Dip the bulb of the flask in the water, remove, turn on the aspirator, and shake the flask. Ice freezes on the outside of the bulb as the liquid boils on the inside. Acetaldehyde is better than ether in that it is soluble in water and, therefore, leaves few vapors in the room. It also has a lower boiling point than ether.

At this point, the location of the light beam depends upon the relative refractive indices of the sample and the reference standard. The beam is then divided by a beam splitter mirror. Each half of the beam falls on a cadmium sulfide photocell. Initially, the reference standard and sample may have either equal or different refractive indices. The recorder is set to the proper zero reading by adjusting the mirror, the manual zero adjustment, and the zero control knob. When this is done, the amount of light falling on each photocell is equal. Any change in the refractive index of the sample liquid causes the beam to shift and more of the light falls on one photocell than on the other. The photocell detector circuit becomes unbalanced and a signal is impressed on the amplifier. The amplified signal energizes the recorder motor, and it drives the beam deflector in the direction that equalizes the light intensities falling on the two photocells. The position of the beam deflector is thus a measure of the refractive index of the sample. Since this is directly coupled to the recorder pen, the recorder indicates differences in refractive index directly.

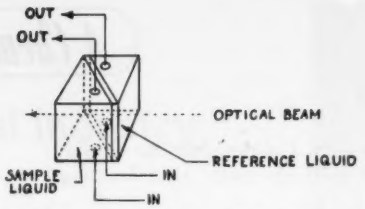

Figure 60. Cell design of the Waters Associates Chromatography Recorder.

Sensitivities as great as $\pm 7 \times 10^{-7}$ in refractive index are obtained; at this sensitivity the range of the instrument is only 0.0003 refractive index units. The cell volume of the instrument described is 0.75 ml. A special chromatographic instrument is available with a flowing reference compartment, similar to the flowing sample compartment. The volume of each compartment is only 0.07 ml. This cell design is shown in Figure 60. The chromatographic refractometer is also equipped with a water-cooled optical bench, a range attenuator, and a strip chart recorder (price: $4500).

Recording differential refractometers similar in design to those described above are manufactured by: Phoenix Precision Instrument Co., Philadelphia 40, Pennsylvania; and Barnes Engineering Co., Stamford, Connecticut.

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Next: Gas Chromatographs.

Note: We are indebted to Dr. H. K. Alber of the Arthur H. Thomas Co. for calling our attention to the following facts: The stainless steel chromatography tank illustrated in Figure 19 (September, 1961, issue) and referred to as the Kensco tank is made by the Arthur H. Thomas Co., and should more properly be designated as the Thomas-Mitchell Tank. Kensco is a distributor, but not the manufacturer, of this item. The hydrometer jar apparatus shown in Figure 23 and attributed to Kensco is also made by A. H. Thomas Co., and is distributed by Kensco. The Chromatobox illustrated in Figure 20 is not manufactured by Research Specialties Co., as stated, but rather is made by Hartnack, Berlin, Germany, and distributed by RSCo.

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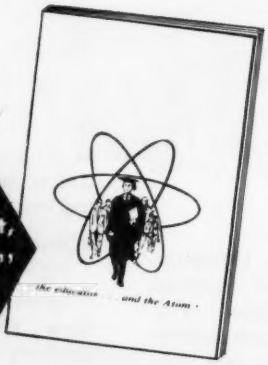

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Fine Weighing Dial: 10 grams by .1 gram graduations
(Readability: .02 g)

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Capacity: 120 grams
Weight-loading Dial: up to 9 grams by 1 gram increments
Fine Weighing Dial: 1 gram by .01 gram graduations
(Readability: .002 g)

TORSION DWL2-1
Specifications are same as the DWL-2 except that this model has scoop for seeds or other bulky material

TORSION DLT5
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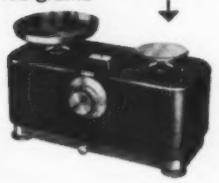
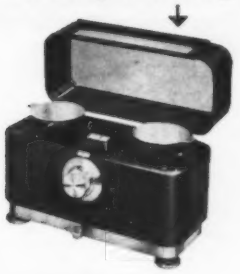
Dial graduated 10 grams by .1 gram graduations
Readability of Dial: .02 grams



TORSION DLT2
Capacity: 120 grams
Dial graduated 1 gram by .01 gram graduations
Readability of Dial: 2 mg



TORSION DLT2-1
Same specifications as DLT2 but has scoop for seeds or other bulky material

TORSION DRx
Capacity: 120 grams
Dial graduated 15 grains by ⅛ grain graduations and 1 gram by .01 gram graduations
Readability of Dial: ¼ dial division

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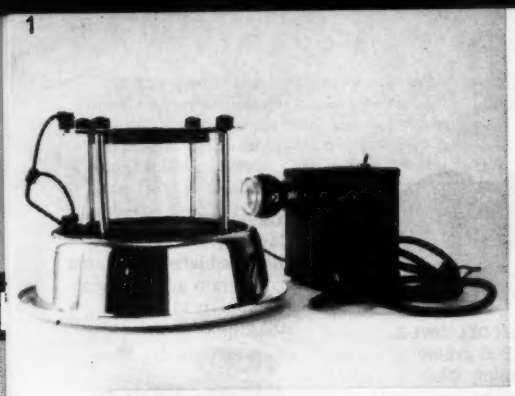

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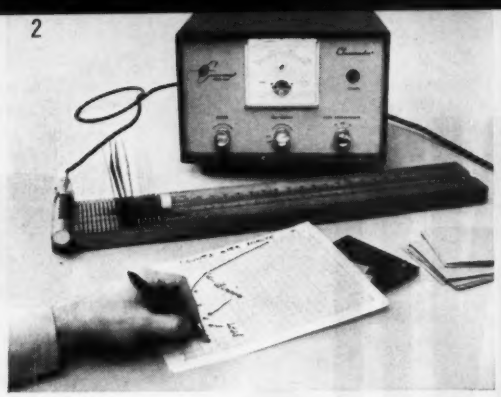

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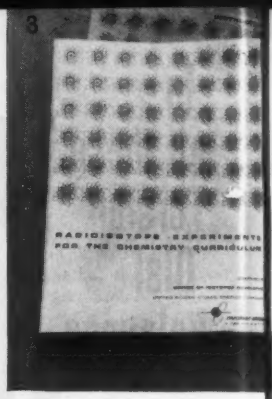

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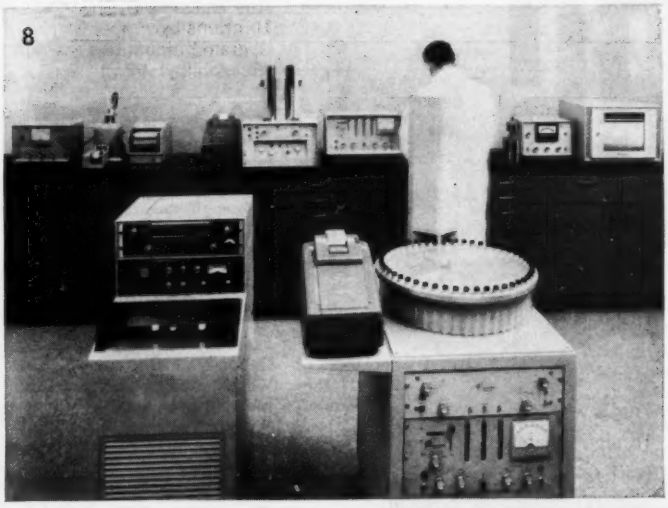

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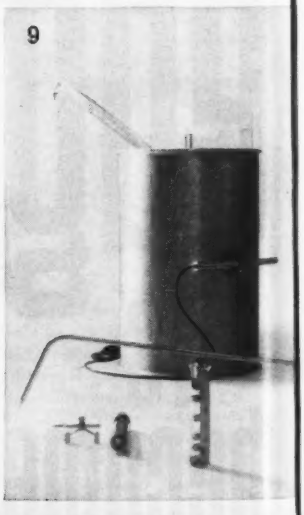

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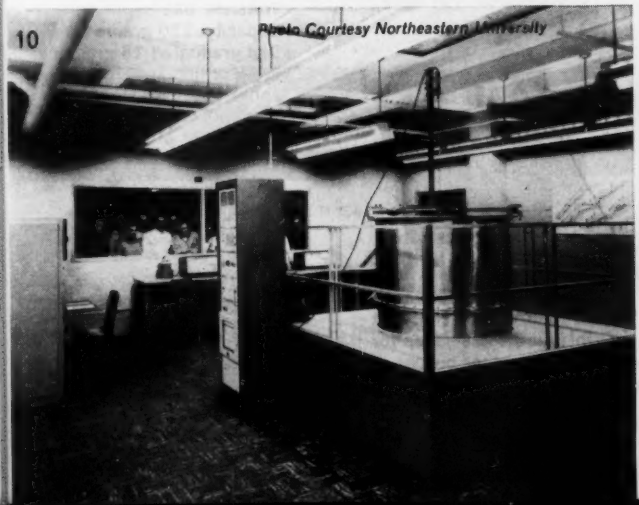

Photo Courtesy Northeastern University

11

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How to bring radioisotopes into your chemistry curriculum

Read how these Nuclear-Chicago products help to enlarge the scope of chemical education and also serve graduate and faculty research needs

RADIOISOTOPE procedures offer chemistry students penetrating insights into the laws of nature. Forward-looking educators are using the equipment illustrated at the left in lectures, for student experiments, and in research. These eleven photographs suggest the scope and completeness of Nuclear-Chicago's instrument line for teaching and research. Systems range from low-cost equipment for demonstration to sophisticated instrumentation for fundamental investigation. All are characterized by reliability, precision, versatility, and inherent safety.

Chemistry departments now employing radioisotope techniques will want to consider program expansion to take advantage of the newer products illustrated. Others may well regard the apparatus presented here as a planning guide.

Low-cost demonstration equipment for the nuclear age became a reality with the introduction of Nuclear-Chicago's Cloudmaster® and Classmaster®. The Cloudmaster (figure 1) is a continuously sensitive cloud chamber that provides a spectacular display of ionization tracks caused by alpha, beta, gamma, and meson radiation. The Classmaster (figure 2) is a complete system for demonstrating radiation absorption, scattering, geometry effects, coincidence loss, etc. In addition, it permits simple tracer experiments using license-exempt radioisotopes. Both instruments enjoy wide acceptance because of their simplicity of operation and the vivid, easily-understood presentations they make possible.

Thirty-three student experiments that fit naturally into the chemistry curriculum without requiring changes in basic course content are contained in a new 187-page experiment manual and 60-page companion volume of instructor notes (figure 3) developed by Nuclear-Chicago under contract with the Atomic Energy Commission. Model 4001 nuclear training system (figure 4) is offered with isotopes, sample preparation equipment, and instruments to perform all the experiments covered in the manual. The system features ease of sample preparation using the chimney funnel shown in figure 5.

Model 4001 is supplied with the long-lived radionuclide set RNS-110 (figure 6) and the radionuclide certificate book RCB-1 (figure 7) with which the instructor can order short- or long-lived isotopes as they are needed. Neither requires an AEC license to purchase or use, and both are available individually.

As student mastery of radioisotope procedures advances, a chemistry department will next want to consider establishing a complete nuclear laboratory for precision handling, detection, and counting of beta- and gamma-emitting isotopes. Such a facility has valuable application to qualitative and quantitative studies at both the graduate and undergraduate levels. A typical installation is pictured in figure 8. In the left foreground is the Model 701 liquid scintillation detector with Logic scaler, and at the right is the Model C120 automatic sample changer for gamma-emitting liquids or solids in test tubes. In the background, from the left, are the Actigraph II® system for scanning radiochromatograms, a low-background automatic sample changer for solid beta samples, and the Dynacon® electrometer for high-efficiency measurement of beta samples in the gaseous phase.

At this stage, the ideally equipped university will lack only the means to create its own radioactive isotopes and to study reactor techniques and fundamental nuclear properties. For these basic needs, Nuclear-Chicago offers a three-step program.

First, undergraduate departments will choose the NH3 neutron howitzer (figure 9) for experiments with low-level neutron sources, production of short-lived, low-intensity radioisotopic tracers, and low-flux activation analysis.

Second, the chemistry, physics, and engineering faculties will often cooperate in establishing an advanced training facility centered on the inherently safe Model 9000 subcritical assembly (figure 10). Characteristics of this water-moderated, natural uranium assembly are nearly identical in nature to those of a full-scale power or research reactor, and the student readily masters the fundamentals of reactor theory and design.

Finally, the long-felt need of university research scientists for a versatile, relatively inexpensive, high-intensity neutron source can be well satisfied by the TNC neutron generator designed and manufactured by Nuclear-Chicago's subsidiary, Texas Nuclear Corporation (figure 11). Providing a controllable yield of 4×10^{10} neutrons per second and a thermal neutron flux of greater than 10^8 using an appropriate moderator, the generator is widely employed in activation analysis, process control, applied research and development, and fundamental investigation.

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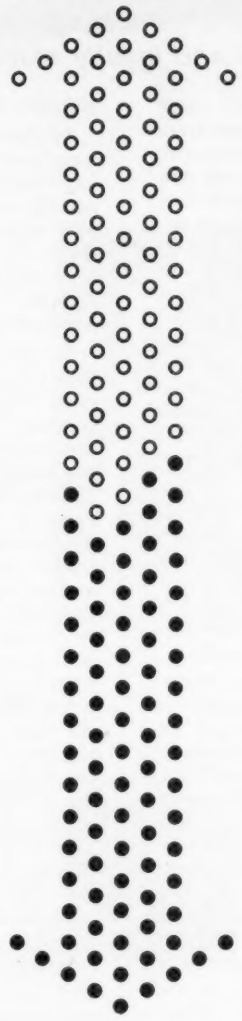

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CHEMICAL PROJECTS

Research Ideas for Young Chemists

Compiled by **JAY A. YOUNG**, Kings College, Wilkes-Barre, Penna.
JOHN K. TAYLOR, National Bureau of Standards, Washington, D. C.

Banding of Basic Mercuric Chloride in Silica Gel

See RUSH, R. I., ET AL., *J. Chem. Educ.*, **24**, 238–9 (1947).

Reactions in gels are interesting to observe; the results are usually esthetically pleasing, and often instructive to study. Of the various reactions which can easily be set up, those involving periodic precipitation are particularly adaptable for investigation by a beginner. Two reagents are selected which will form a precipitate when their solutions are mixed. One of these is mixed with a solution of sodium silicate, then silica gel is formed by the addition of an acid. A solution of the second reagent is poured over the top of the gel and in a short time a precipitate forms within the gel. Depending upon the concentrations of the two reagents, the temperature, the *p*H of the gel, the nature of the gel (gels other than silica gel can be used), and upon other factors, the precipitate will often form in bands with clear volumes of gel between each band of precipitate. With only reasonable care, the results are reproducible but, as yet, no completely satisfactory explanation of the formation of successive bands of precipitate has been proposed.

Other articles which deal with this phenomenon are: FILLINGER, H. H., ET AL., *J. Chem. Educ.*, **22**, 440–1 (1945) and FILLINGER, H. H., *J. Chem. Educ.*, **25**, 102–3 (1948).

Question:

From the results of your own study in the laboratory and from your reading on the subject, propose an explanation for the phenomenon of periodic precipitation in gels and test the validity of your explanation by further laboratory work.

The fact that experienced investigators have not yet solved the problem posed should not discourage a student from his study of the same problem; rather, it should whet his interest further. For the past several hundred years, many problems in science have been solved by amateurs, and there is no reason to suggest that such an individual can no longer make a significant contribution.

Bredig Sols: A Lecture Demonstration

See REIGEL, E. R., ET AL., *J. Chem. Educ.*, **26**, 51–920 (1949).

Metals can be dispersed in a liquid to form a stable colloidal suspension by striking an electric arc between metallic electrodes under the surface of the liquid. An arc welder which delivers direct current to the welding electrodes can be used as the source of current. Details are given in the article.

Questions:

If the two electrodes are made of different metals, are the micellae which form different than when both electrodes are made of the same metal?

If the electrodes are alloys instead of pure metals, are the micellae formed different? If so, in what ways?

Upon what factors does the charge on the micellae depend?

Are the micellae you produce pure metals or a compound, or both? Can you produce either metallic or compound micellae by varying the conditions of formation?

Can the suspensions you produce be made stable for very long periods by the use of protective colloids? If so, which such substances are most efficient?

Can Bredig's method be modified to produce sols of nonconducting substances?

If flammable liquids are used, take care to strike an arc only when the electrodes are completely immersed in the flammable liquid. A supply of flocculent asbestos should be on hand, to cast over the surface of any burning liquid. All work should be performed with proper precautions taken to avoid electric shock; if in doubt, consult your local electric power company rather than the owner of the borrowed arc welder.

Interference Films on Tungsten

See PARSONS, I. H., *J. Chem. Educ.*, **24**, 483 (1947).

A thin film of oxide can be formed on a disc of tungsten when the tungsten is used as the anode in the electrolysis of a 0.06 *N* sodium hydroxide solution, with an iron disc as the cathode. As electrolysis proceeds, the film of oxide becomes thicker and various colors are observed in succession; when the circuit is broken, the oxide film dissolves, and the same colors appear in reverse order. The process can be halted at any desired moment by removing the tungsten from the electrolysis vessel; the dried film of oxide is stable, and preserves its color.

Questions:

The thickness of the oxide film on stainless steel can be increased by heating this alloy in air. If carefully performed, this process also yields permanently colored oxide films of a desired color. Is it possible to form oxide films of various colors on stainless steel by controlled anodic oxidation?

Can films of oxide, or other composition, be formed on any other metals by an electrolytic process? If so, do any of these films show colors by interference?

By laboratory measurements, show that there is a relation between the color of a film and the thickness of the film. For an unusual way to measure the thickness of a film, see NELSON, L. S., *J. Chem. Educ.*, **28**, 103 (1951).

Cytoplasm-Particle Linked Enzyme Reaction: A Laboratory Exercise

See LITWACK, G., *J. Chem. Educ.*, **35**, 358–9 (1958).

The cytoplasmic fluid obtained from the homogenization of fresh chilled rat liver can be separated, by centrifugation, from the mitochondria and other cellular products. The cytoplasmic fluid contains an enzyme, xanthine oxidase. Another enzyme, uricase, remains with the mitochondria.

Xanthine, a substrate, is also present in the cytoplasmic fluid, and is oxidized in the presence of xanthine oxidase to the enol form of uric acid. An equilibrium between the reduced form, xanthine, and the oxidized form, uric acid, is established.

The enol form of uric acid isomerizes to, and is in dynamic equilibrium with, the keto form.

But, in the residue from the centrifugation, with the mitochondria, is a second enzyme, uricase, which will oxidize the keto form of uric acid to allantoin (and probably other products).

The presence of xanthine can be determined colorimetrically, and, if the two separated portions of the centrifuged homogenate are mixed, a decrease in the quantity of xanthine should be observed, because the uric acid, in equilibrium with xanthine, will be further oxidized in the presence of uricase. This effect is observed, and the procedural details for the study of the linked enzyme, xanthine oxidase-uricase, system are described in this article.

Questions:

Show that xanthine oxidase and uricase form a linked enzyme system.

Is it true, as suggested by Litwack, that xanthine dehydrogenase migrates with the globulin fraction in paper electrophoretic separations? (See GLENN, W. G., AND JAEGER, H. A., *J. Chem. Educ.*, **35**, 360–1 (1958) for a description of the construction of a paper electrophoresis apparatus.)

Litwack suggests that it may be true that xanthine oxidase is loosely bound to lipoprotein or fat bodies at the surface of the mitochondria. Can you find evidence to support this suggestion?

There are many other linked enzyme systems, some with more than two enzymes; show, by a series of experiments similar to those described by Litwack, that a linked enzyme system is present in a biochemical milieu which you select for study.

A Laboratory Demonstration of Blood Catalase

See BLACKWELL, R. Q., AND FOSDICK, L. S., *J. Chem. Educ.*, **32**, 588–90 (1955).

Catalase, an enzyme which is present in blood, catalyzes the decomposition of hydrogen peroxide to water and oxygen. This decomposition is exothermic, and comparative measurements of the temperature rise of buffered solutions of hydrogen peroxide, to which catalase-containing substances are added, can be used to determine the catalase activity of these substances. For calibration, standardized catalase solutions should be used; these are commercially available.

Blackwell and Fosdick describe, in complete detail, a procedure for the determination of the catalase activity of human blood. Their procedure requires the use of 30% hydrogen peroxide; this liquid should not be handled carelessly. Before using it determine the safety precautions that are necessary and follow them during your work.

Questions:

Show that the catalase activity of blood withdrawn from several human subjects is, or is not, an indication of the state of health of the subjects.

Many animal tissues and many plant tissues also contain catalase. After removing other catalysts which might catalyze the decomposition of hydrogen peroxide, show that a sample of plant or animal tissue does or does not contain catalase.

Does the catalase content of various tissues from the same plant vary from place to place in the plant? If so, suggest a reason and test your explanation by further investigations.

Papain as an Enzyme Catalyst in Undergraduate Organic Chemistry

See ABERNETHY, J. L., AND KIENTZ, M., *J. Chem. Educ.*, **36**, 582–4 (1959).

Many enzymes are not suitable for elementary investigations of the characteristics of enzymatic action because they are destroyed or inhibited by variables which are difficult to control. Of those which can be studied easily, papain is one of the best; it is readily available, easily activated, inexpensive, and can be stored under refrigeration for long periods.

Papain catalyzes the hydrolysis of amide linkages, of other peptide-like bonds, and of esters of amido acids. Like any other catalyst, it also catalyzes the reverse reaction. Abernethy and Kientz list several interesting examples of this catalytic action. The reaction of hippuric acid and aniline to form hippuric anilide, the separation of racemic mixtures of amino acids, the preparation of diacylhydrazines, and other applications, are discussed. Details are not given; instead, the pertinent references are cited from which the necessary information can be obtained.

Since a large number of investigations are suggested in this article, no questions need be appended here.

The Laboratory Preparation of a Simple Vitamin: *p*-Aminobenzoic Acid

See KREMER, C. B., *J. Chem. Educ.*, **33**, 71–2 (1956).

This vitamin can be prepared, starting with toluidine, by acylation, oxidation of this product to *p*-acetaminobenzoic acid, and hydrolysis to *p*-aminobenzoic acid. The apparatus required is readily available and the synthesis is straightforward; all necessary details are given.

Questions:

Prepare *p*-aminobenzoic acid, starting with a compound simpler than toluidine, such as toluene, or, more difficult, benzene.

Prepare *p*-aminobenzoic acid by a procedure which is distinctly different from that given in this article.

Can *p*-aminobenzoic acid be obtained from natural sources?

Is *p*-aminobenzoic acid an essential constituent of the diet of white rats, hamsters, Drosophila melanogaster, or any other experimental animal which can be easily maintained in a controlled environment? If so, what role does it play in the metabolism of these animals?

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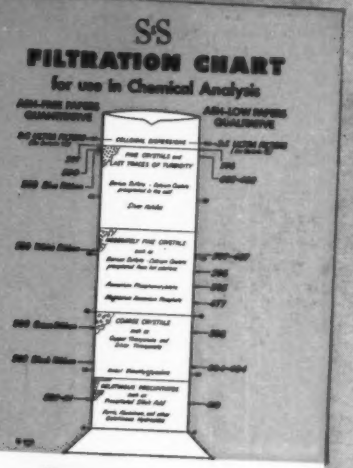

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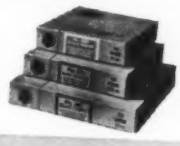

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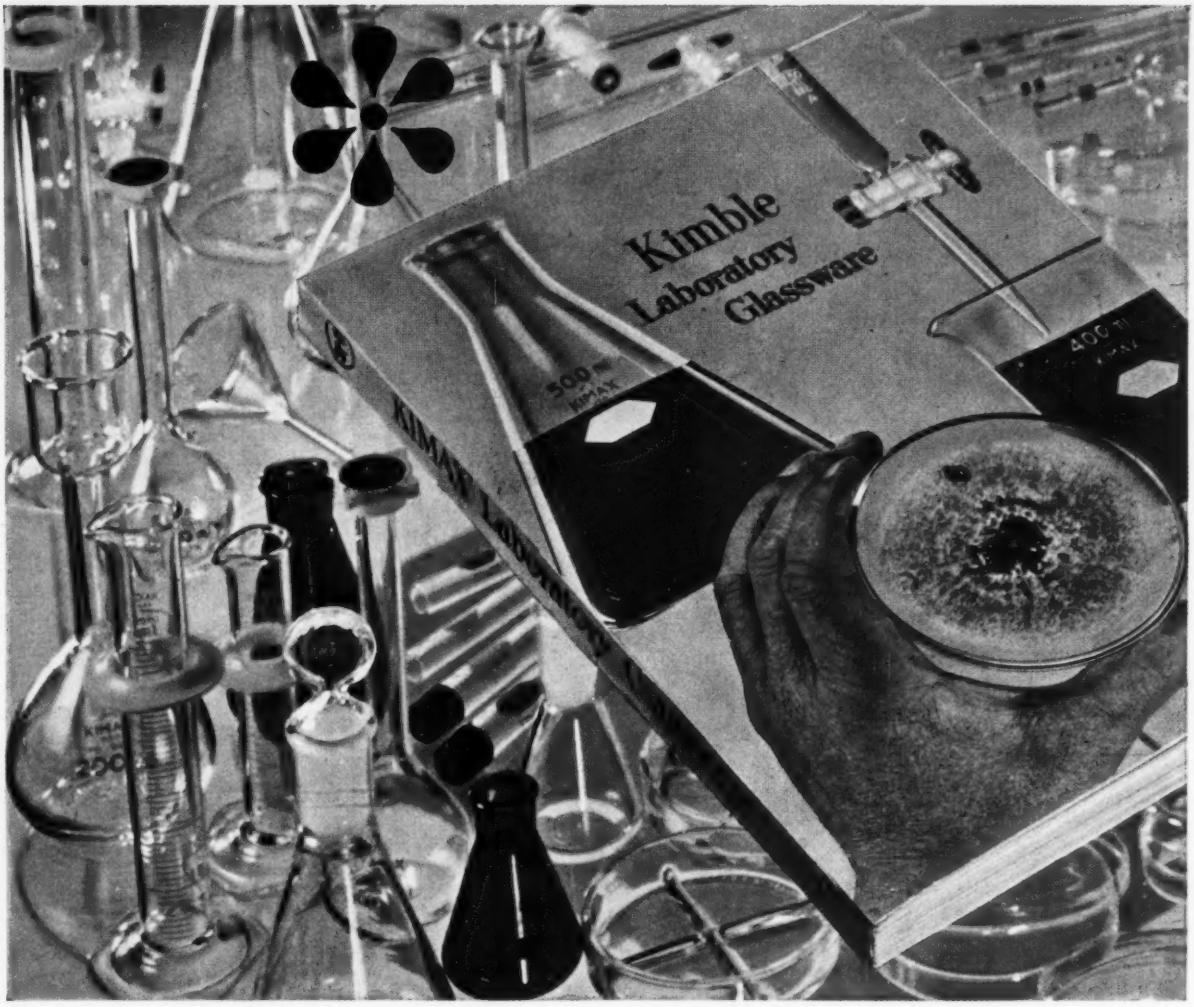

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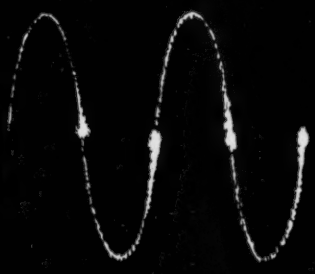
SEE 

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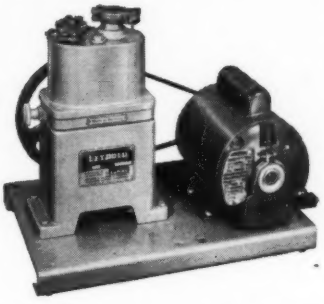

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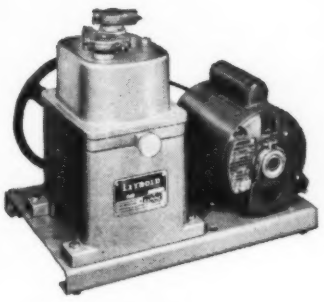

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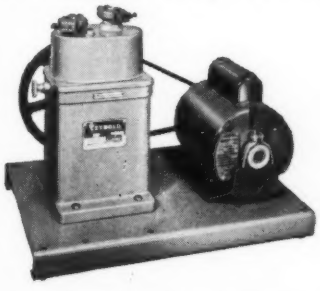

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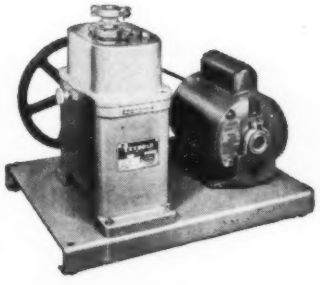

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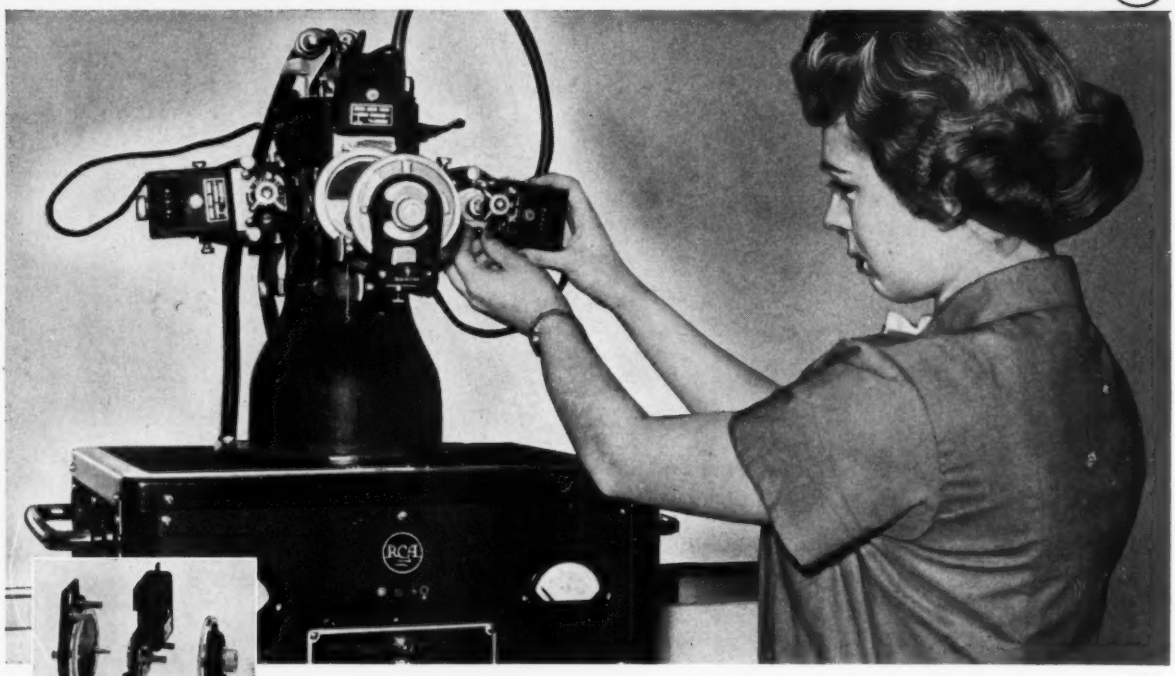

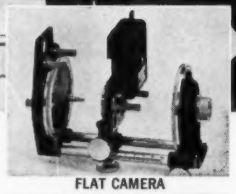
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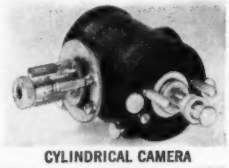
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For a folder giving more complete description and prices, write to Alyea-Jackman Kits, Inc., P. O. Box 666, Pottstown, Pennsylvania.

▶ A new, low-cost vacuum pump for educational and industrial applications was announced recently by Atomic Laboratories, Inc., 3086 Claremont Ave., Berkeley 5, California. Called the V-2, the pump is of rotary design and can achieve vacuums down to 0.15 mm. of mercury. Its low cost is due to a new two-stage evacuating system which eliminates nearly half the moving parts found in conventional pumps.

▶ A newly designed pipettor that can be set accurately for any capacity within the range of 2 to 25 ml. has just been introduced by the Manostat Corp., 25 N. Moore St., New York 13, N. Y. This Macro Accropet, latest addition to the Manostat family of precision pipettors, permits the user to employ one precise setting repetitively, without error, until the Accropet is reset.

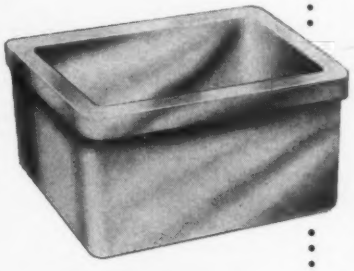
▶ Development of remote handling tongs with new continuous locking-action and parallel motion jaws is announced by Ameray Corp., 1003 Route 46, Kenvil, New Jersey. Use of this equipment promises safer handling of explosives, radioactive substances, and toxic materials. These relatively inexpensive tongs can do almost anything that can be done by hand, including: lift beakers, handle "hot" material, flick switches, pour fluids, and mix chemicals.

▶ Bausch & Lomb's Balomatic slide projectors can now be completely operated from 30 feet away. B&L's announcement of a new remote control cord, doubling the length of the current 15 foot cord, will make the Balomatic even easier to use for large group slide shows. Contact Bausch & Lomb, Inc., Rochester 2, New York, for further information.

▶ A new Swing-down emergency eye, nose and mouthwash fountain for laboratories, that offers first-aid for facial contamination and the effects of acids, chemicals, "hot" fuels, caustics, radiological, bacteriological and other foreign matter, is now available from Logan Emergency

(*Continued on page A831*)

"U. S." CHEMICAL PORCELAIN LAB SINKS



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Available in three attractive colors to match any decor — cool "surf green," soft "mist gray" and sparkling white — these non-staining, scratch-resistant one-piece sinks are as easy to clean as a china dish — no scouring or honing is ever required. "U.S." Chemical Porcelain laboratory sinks can be supplied in many types and sizes. For example, in the Fig. 108 model, 46 sizes are available.

For complete details contact your Laboratory Furniture Manufacturer or write direct for free Bulletin L-10.

Chemical Ceramics Division

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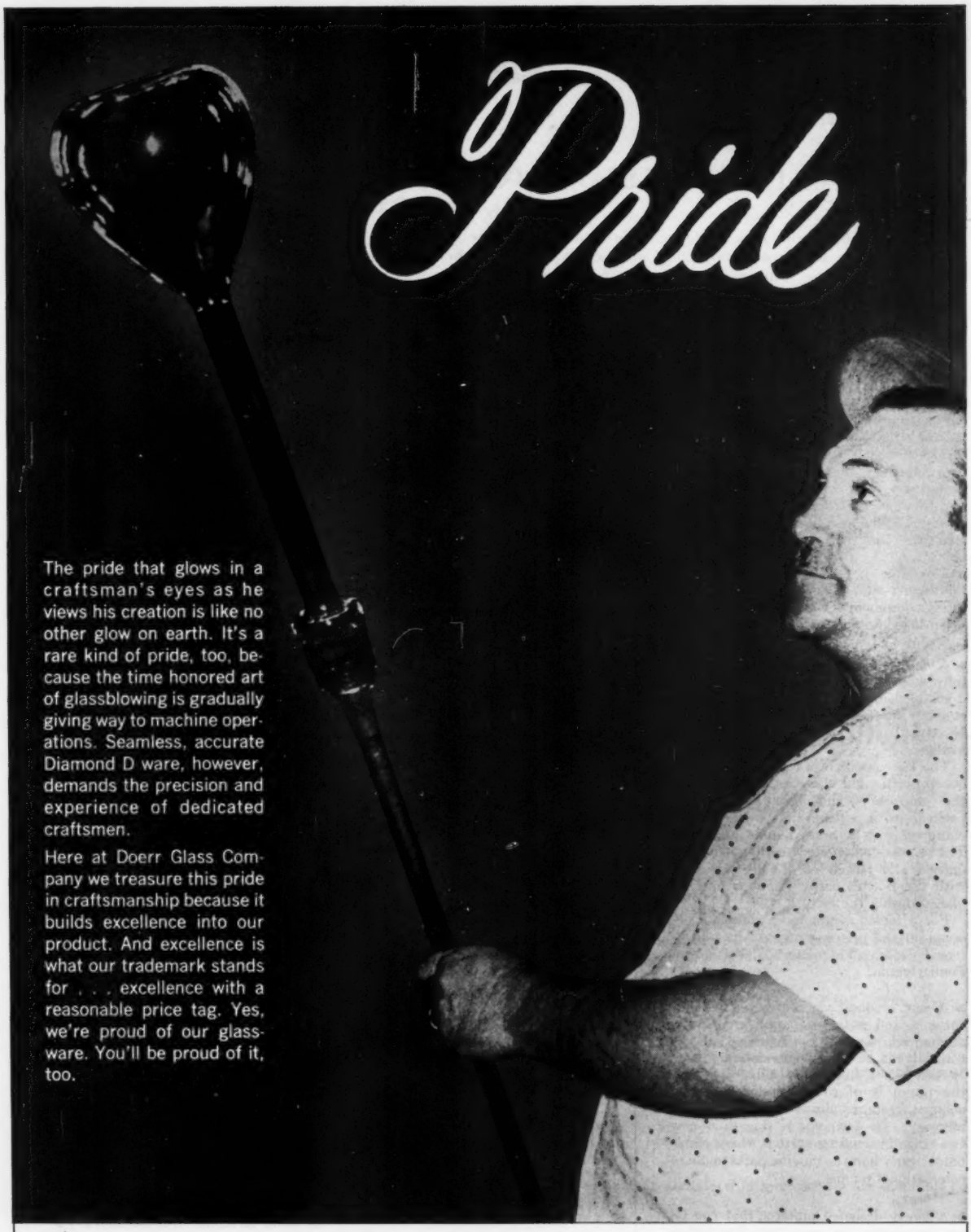
AKRON 9, OHIO

B2-H



The pride that glows in a craftsman's eyes as he views his creation is like no other glow on earth. It's a rare kind of pride, too, because the time honored art of glassblowing is gradually giving way to machine operations. Seamless, accurate Diamond D ware, however, demands the precision and experience of dedicated craftsmen.

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Out of the EDITOR'S BASKET

Showers, Inc, P. O. Box 111, Glendale, California.

▶ Labline, Inc., 3007 W. Grand Ave., Chicago 22, Illinois, has introduced a brand new development in controlled laboratory shaking with its new Hot Air Shaking-Incubator, Model 3570. Available in regular and king size which holds up to 200—20 ml. beakers or 162—25 ml. Erlenmeyer Flasks. Temperature range is up to 65°C. . . . Uniformity within + or −0.5°C.

▶ Radiation Equipment & Accessories Corp., Lynbrook, New York, has developed an assortment of inexpensive interchangeable phosphors. The unit, called the E-160 "Scinti-Kit," can be used with scalers for the measurement of alpha, beta, or gamma emitting nuclides, or with ratemeters as an all-purpose laboratory monitor.

▶ The tricky problem of preparing reproducible radioactive samples, which plagues most students, has been greatly simplified by a new sample preparation set, the Model PM-5, developed by Nuclear-Chicago Corp., 359 E. Howard Ave. at Nuclear Dr., Des Plaines, Illinois. The sample preparation set employs a newly-developed filtration device called a chimney funnel which contains a porous stainless steel filter disc. A solution is suction filtered through the chimney funnel and a uniform layer of sample is deposited and dried on the steel disc.

▶ Hoover Melting Point Apparatus, "Uni-Melt," which is rapidly becoming a standard instrument for determination of capillary melting points, has recently been improved to overcome fluctuation in laboratory lighting. Ask for *Bulletin 133*, Arthur H. Thomas Co., P. O. Box 779, Philadelphia 5, Pennsylvania.

New Literature

● The Varniton Co., 416 N. Varney St., Burbank, California, offers a bulletin on *Varniton Jet Mixers for the Laboratory.*

● Sargent *Bulletin D* describes in detail the new Sargent Recording Titrator and is now available from the designer and manufacturer, Dept. RT, E. H. Sargent & Co., 4647 W. Foster Ave., Chicago 30, Illinois.

● A new brochure reporting the fast growing chemical operations of W. R. Grace & Co. is available upon request to the Public Relations Dept., W. R. Grace & Co., 3 Hanover Sq., New York 4, N. Y.

● The 1961 edition of *Hooker Chemicals*, a 16-page bulletin *100-E* listing and describing the firm's 108 organic and inorganic chemicals for industry and agricul-

(*Continued on page A832*)

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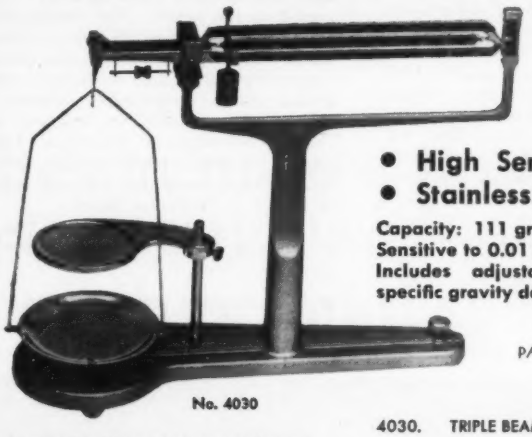

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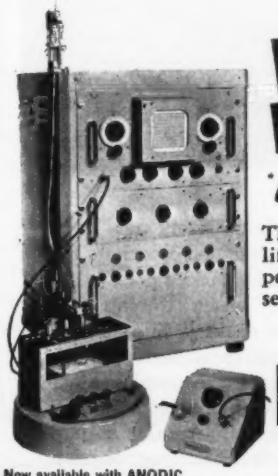
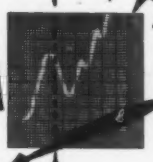


A COMPLETE POLAROGRAM



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ture, is now available from Hooker Chemical Corp., Niagara Falls, New York.

● Copies of new 1008-page general *Catalogue J-300* are available from Central Scientific Co., a division of Cenco Instruments Corp., 1700 W. Irving Park Rd., Chicago 13, Illinois. Requests should be made on a company or official letterhead, and the individual making the request should give the research, manufacturing or educational activity at his location.

● A new technical bulletin containing a complete list of all currently available chemical-loaded molecular sieve products has been released by Linde Co., Division of Union Carbide Corp.

The listing includes the products which are obtainable in experimental and commercial quantities and classifies them, according to the type of chemical that is loaded on the sieves. The classifications are: primary amines, secondary amines, tertiary amines, polyamines, metal organic compounds, peroxides, acids and anhydrides, sulfur compounds, antioxidants and plasticizers, alcohols, halogens and various miscellaneous materials. Information is provided also on the various types of compounds that generally cannot be loaded on Molecular Sieves.

Copies of the bulletin, *F-1514*, may be obtained by writing Linde Co., 270 Park Ave., New York 17, N. Y.

● A new section of the *6-C Catalogue*, covering steel laboratory equipment and furniture, has been published by the manufacturer, Metalab Equipment Co., Duffy Ave., Hicksville, L.I., New York. Write for a copy.

● The plasticizing of resin coatings is covered from acrylics to zein in a new, 54-page technical bulletin, *PL-327*, published by Monsanto Chemical Company's Organic Chemicals Division. Contact Mr. Robert Hogan, Organic Chemicals Division, Monsanto Chemical Co., 800 N. Lindbergh Blvd., St. Louis 66, Missouri, for copies.

● College Science Publishers, P. O. Box 798, State College, Pennsylvania, offers *The Pesticide Handbook* which lists the names, active ingredients and manufacturers of over 9000 insecticides, fungicides, rodenticides, herbicides and other agricultural chemicals and equipment. It also contains valuable information on current legal tolerances for all pesticides, as well as a general discussion of the uses, compatibilities, and antidotes for all pesticides, both old and new. The editor is Dr. Donald E. H. Frear, well-known authority on the chemistry of pesticides, and the price is $2 per copy paper bound, $3.50 per copy cloth bound.

● New 936-page catalogue of laboratory supplies, equipment, and instruments has just been published by LaPine Scientific Co. Thousands of laboratory items are clearly illustrated and concisely described. Descriptions include specifications that are complete enough to enable purchasing directly from this catalogue.

Items are listed alphabetically by names in common usage, and catalogue numbers follow the alphabetical listing by means of an ascending numbering system. The combination of these two systems makes this catalogue fast and easy to consult. A comprehensive, extensively cross-referenced subject index pinpoints the location of every item.

Handsome, hard-cover catalogues are free to persons requesting copies on commercial or institutional letterheads. Write to LaPine Scientific Co., Catalogue Dept. 6001 So. Knox Ave., Chicago 29, Illinois.

● The Cobalt Information Center, c/o Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio, offers a news brief which lists literature currently available from the Center.

● Radiochemists, crystallographers, chemists, and physicists will be interested in the Pergamon Press, Inc., 122 E. 55th St., New York 22, N. Y., new publication *Radiochemistry U.S.S.R.* Contact their office for details.

● Ottenheimer Publishers, Inc., 4805 Nelson Ave., Baltimore 15, Maryland, offers, *Trig, Log, Antilog and Log of Trig Functions 6 Place Tables for $1.*

● The *Periodical Monitor*, a new magazine, provides engineers immediate reference to hundreds of articles on electronics and instrumentation each month. It features abstracts of the articles and is cross-refer-

(*Continued on page A834*)

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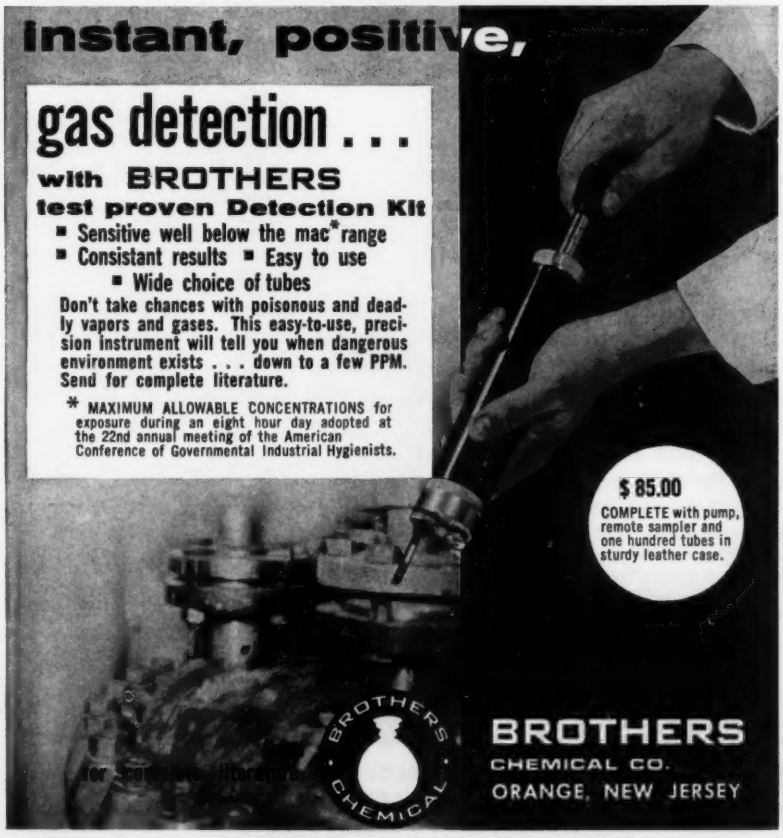
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enced, with every abstract listed under every possible subject heading. There is no advertising content.

The publisher, The Periodical Monitor and Abstract Service, 15 N. Euclid Ave., Pasadena, California, will send subscription information and illustrated brochure upon request.

● *Properties and Essential Information for Safe Handling and Use of Methyl Ethyl Ketone* is the title of the latest chemical safety data sheet, SD-83, now available from the Manufacturing Chemists' Association, Inc., 1825 Connecticut Ave., N. W., Washington 9, D. C., for 30 cents.

● The awareness that close group effort is required to further space technology is inherent in *Ballistic Missile and Space Vehicle Systems* just published by John Wiley & Sons, Inc., 440 Park Ave., South, New York 16, N. Y. (526 pages, $12). "It is quite clear that for weapon systems and space vehicle systems it will be necessary to extend every discipline to its limit" and that their integration is necessary from the ground up, the book states at the start. A multiple authorship of twenty-two specialists in the field, including the editors—Howard S. Seifert and Kenneth Brown—underscores this approach.

● Order the following, by number, from the O.T.S. of the Business and Defense Services Administration, U. S. Department of Commerce, Washington 25, D. C.

AEC-tr-4432. *Radiometry*, published in Moscow, 1956, $4.50. PB 161 887. *Literature Survey on Research and Development on High Pressure Technology.* L. Berg and H. Herman, Engineering Supervision Co., for Wright Air Development Division, U. S. Air Force. March 1960. 54 pages. $1.50. TID—6613. *Radioisotopes in World Industry—Abstracts of Selected Foreign Literature.* January 1961. $2.50. PB 171 376. *The Thermochemistry and Photochemistry of Organic Halogen Compounds.* R. Burkhart and others, University of Colorado, for U. S. Air Force. September 1960. 98 pages. $2.25. PB 171 155. *Second Status Report on Fuel Cells.* B. R. Stein and E. M. Cohn, Office of the Chief of Research and Development, Departmentof the Army. December 1960. 80 pages. 75 cents. SB—439 *Machine Translation.* Reports of the Army, Air Force, and other Government agencies, as well as foreign research literature, are listed pertaining to such aspects of machine translation as information evaluation, study of Zipf's law, digital encoding of Russian scientific text, inductive inference, retrieval, electronic computing, and word lists. 10 cents.

● A three volume compilation of approximately 150 selected papers published by the National Bureau of Standards on precision measurement, calibration and related subjects is available from the Superintendent of Documents, Government

(*Continued on page A836*)

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Printing Office, Washington 25, D. C.: Volume I—*Electricity and Electronics.* Catalogue No. C 13.11:77/v.1, cloth, $6.00. Volume II—*Heat and Mechanics.* Catalogue No. C13.11:77/v.2, cloth, $6.75. Volume II—*Optics, Metrology, and Radiation.* Catalogue No. C 13.11:77/v.3, cloth, $7. Set of all 3 volumes—$19.75.

● *A Spectrophotometric Atlas of the Spectrum of CH from 3000 A to 5000 A*, by Arnold M. Bass and H. P. Broida, National Bureau of Standards Monograph 24, issued February 14, 1961, 20 pages, 20 cents. (Order from Superintendent of Documents, Washington 25, D. C.)

Miscellany

★ Michael Faraday, when a member of a special royal commission, investigating the state of pictures in London's National Gallery in 1850 said, "I have not the smallest doubt that a person of competent chemical knowledge and a little acquainted with the practice of painting in ancient and modern times might be valuably employed in ascertaining points on the preservation of pictures by means of varnishes or other things . . . and I wonder that it should be left, as it were, to accident when accurate knowledge upon the subject might be so easily acquired."

Today, according to "Art's Silent Partner's" in *The Laboratory*, Volume 29, No. 3, The Fisher Scientific Co. publication, 133 museum laboratories on 6 continents are really solving many of the artist's problems.

★ Three new gas-in-metal standard samples—of titanium containing three different levels of hydrogen—are now available from the National Bureau of Standards. These reference materials, developed by J. T. Sterling and L. L. Wyman of the Bureau's chemical metallurgy laboratory, are part of a series initiated in 1959 to provide standards of gases-in-metals for a variety of metallurgical products. Such standards are suitable for calibrating chemical analytical equipment employed to measure the gas content in various alloys. Standard samples issued by the Bureau are described in Standard Materials, *NBS Circular 552* (third edition), which may be obtained for 35 cents from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Up-to-date supplementary inserts, which are issued periodically, are available upon request directly from the National Bureau of Standards.

★ The Thirteenth Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, sponsored by the Analytical Chemistry Group of the Pittsburgh Section of the American Chemical Society and the Spectroscopy Society of Pittsburgh, will be held at the Penn-Sheraton Hotel in Pittsburgh, Pennsylvania, from March 5 to March 9, 1962.

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Mighty magnification

Forty bucks for a few rings and rods to put over the front of the new *Kodak Retina Reflex III Camera?**

A mite under, actually, at the camera shop. Also includes an auxiliary lens. All nicely fitted together. Called the Kodak Retina 1:1 Copying Kit. *Switch the f/2.8 lens from the camera to the kit, snap the kit onto the camera, attach the auxiliary lens and the camera's lens hood, set the diaphragm between f/11 and f/22 and the distance scale at 5 feet. Snap, swish—just like that. Make pictures. Anything in the plane of the rectangular opening is focussed on the film same size. Depth of field at f/22 is 5.5 mm. When resulting slide is projected, magnification is mighty and can be useful for mensuration if a calibrating scale has been included in the picture.*

If you can make this kit yourself for less than $40, you are too clever a fellow to be working that cheap.

*Most versatile 35mm camera we make. Also works with *Kodak Retina Reflex S* and *IIIS Cameras*. Kit designed for f/2.8 models only.

A sharp eye for infrared

The decision to announce at this time the availability of *f*/1 *Irtran-2 Aspheric Lenses* has been reached in struggle against deeply rooted inhibitions. In the photographic film and paper trade we are habituated to a longer silence before the first blast of the trumpets. Infrared technology hates to wait, however.

These Irtran-2 lenses transmit usefully from 2μ to 14μ. Two focal lengths, 2-inch and 3-inch, are offered off the shelf. At *f*/1, we seem to have done well at providing high collecting-power for energy without undue sacrifice of sharpness. Sharpness was the goal. For both lenses, the minimum circle of confusion *computes* at less than .001″ for any wavelength from 4.25μ to 10μ.

In the 1μ-3μ region, the sharpness does not compute to be as good as farther out in the infrared. Yet we have customers who use the lenses there and are happy with confusion-circle minima as large as .008″.

In comparison with reflective optics hitherto used, Irtran-2 aspheres offer compactness and a wider field that doesn't even show any appreciable deterioration as far as 2° off axis. You do give up the perfect achromatism of reflective optics.

We have said enough to establish our frankness. If impressed and interested, write Eastman Kodak Company, Special Products Division. Bear in mind that Irtran-2 material has a hardness of 354 Knoop, is not at all brittle, withstands thermal shock, doesn't dissolve in water or common organic solvents, and can get very hot without losing transparency or giving off toxic fumes.

The carboxamide way to solvation

A good salesman sells effect harder than he sells cause. The principle works broadly but not universally.

Suppose the effect you seek is solvation. Everybody at one time or another seeks solvation. Therefore we sell laboratories solvation, among other things. The invoice, to be sure, is made out for some solvent but solvation, in these cases, is what's really being bought. In these cases, our business is no different from most other businesses. There are other cases where our business is peculiar. *N,N-Dimethylbenzamide* (Eastman 8417) typifies the peculiarity.

Eastman 8417 is probably a most potent solvent, but when you buy it you are just buying N,N-dimethylbenzamide. Should it provide the solvation you seek, count yourself lucky. If we were to put squads of chemists to work fully exploring the properties of each of some 3900 Eastman Organic Chemicals, the enterprise would collapse and that would be a pity.

N,N-dimethylbenzamide joins a class of compounds long noted as "exotic" solvents. Its older cousins, *N,N-Dimethylformamide* (Eastman 5870) and *N,N-Dimethylacetamide* (Eastman 4972), possess an uncanny solvent power for high polymers, organometallics, and inorganics that "ordinary" solvents can't budge. They make excellent vehicles for ionic reactions.

Solvation virtually demands the liquid state. Solubility also usually increases with temperature. Without the trouble and peril of high-pressure tactics, N,N-dimethylbenzamide can be maintained as a much hotter liquid than its cousins. It doesn't boil until 272-273C, compared with 152-154C for N,N-dimethylformamide and 165-166C for N,N-dimethylacetamide. When it is made as pure as we make it, it doesn't even melt until it gets to 42C—a solvent you buy as white crystals. (A putative solvent, rather.) $2.75 buys 5 grams of it that you can use to check our surmise. (Don't take fright. Some day it may move in tank cars.)

We also offer 17 other N-substituted carboxamides that are probably swell solvents.

We also offer Eastman Organic Chemicals List No. 42, which itemizes some 3900 more compounds possessing merits that purchasers can generally be expected to know better than we. For a copy, write: Distillation Products Industries, Rochester 3, N. Y. (Division of Eastman Kodak Company).

Prices subject to change without notice.

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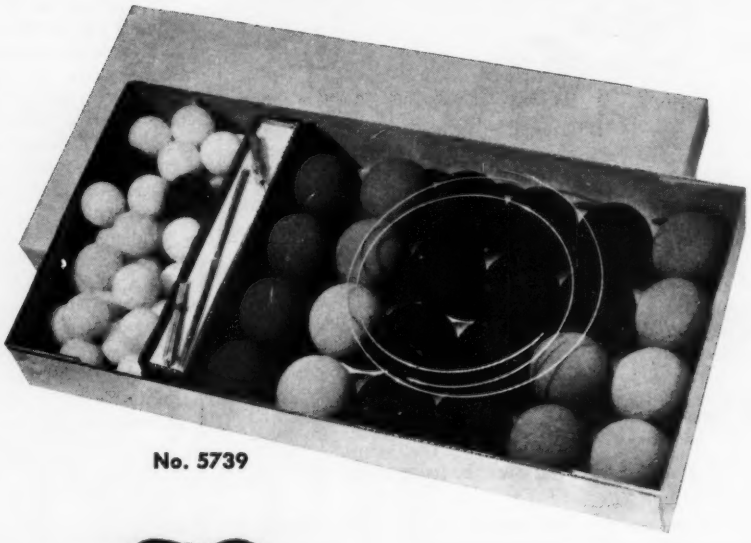
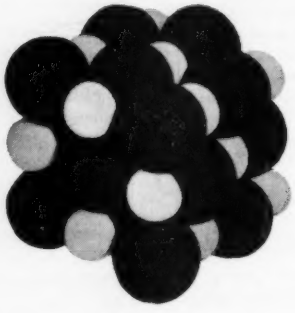
This is another advertisement where Eastman Kodak Company probes at random for mutual interests and occasionally a little revenue from those whose work has something to do with science

Welch Crystal Lattice and Molecular Model Kit



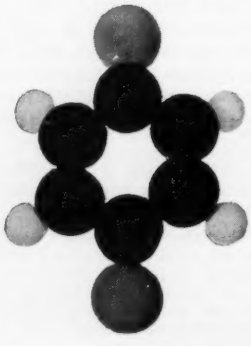
No. 5739

Crystal Lattice of Sodium Chloride →



Differences between covalent and ionic bonding may be demonstrated

Paradichlorobenzene molecule



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EDITORIALLY SPEAKING

A publishing enterprise which should be known to every teacher of college chemistry is the "Monographs for Teachers" series issued by the Royal Institute of Chemistry in London, England. The project was undertaken because

> . . . concise and authoritative accounts of selected well defined topics . . . are needed . . . for the guidance of those who teach chemistry at the General-Certificate-of-Education-Advanced-Level and above. [This is roughly equivalent to American underclass college courses.] . . . This guidance is needed where there are difficulties in presenting a topic clearly in terms of simple fundamental principles and on a basis that would enable the pupil to go on to more advanced studies without having to "unlearn."

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No. 5. *Principles of Chemical Eqilibrium*, by P. G. Ashmore, Cambridge (49 pages, 4*s*. 6*d*., $0.65). The author claims that his purpose is to ". . . show that there is a property characteristic of each substance, and of P and T, the free energy which permits the computation of equilibrium constants." The highest praise we can think of is that he accomplishes his purpose. This book is a little Denbigh.

We congratulate Dr. F. W. Gibbs, under whose editorial direction the series is taking shape, and urge readers to join in the effort by securing copies (postpaid at the prices quoted; send check, bank draft, or money order) from the Royal Institute of Chemistry, 30 Russell Square, London W. C. 1, England.

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Elements of Order-Disorder Theory

Applications to gas adsorption, binary solutions, alloys, and ferromagnetism

This article attempts to present the basic concepts of order-disorder theory in as elementary a manner as seems consistent with the subject under discussion. Toward this end, rigorous derivations have been replaced with heuristic arguments and drawings substituted for mathematical derivations. The treatment is incomplete and obviously slanted in the direction of interest to the writer. Nevertheless it is hoped that the exposition will serve as a primer for readers not already acquainted with this topic.

Broadly speaking, order-disorder theory as used here is concerned with the specification of the statistical and thermodynamic properties of assemblies in which a single constituent can exist in either of two distinct states or in which two separate components are present, each in a single state. Special importance is attached to the role of mutual interactions among constituent particles in the system. The general area of utility will become clearer in the course of the development; applications will be made in the fields of gas adsorption, binary solutions, binary alloys, and ferromagnetism. The diversity of phenomena which can be treated by a single formalism attest to the power and importance of order-disorder theory.

The present development leans heavily on intuitive arguments; therefore it is recommended that for a proper exposition to the subject matter readers turn to several articles by Hijmans and de Boer.[2] These writers present a lucid account of the theory on a more advanced mathematical level. The subject has also been reviewed in a recent article by Domb.[3]

To facilitate the general presentation we begin with the following sample problem: Consider the surface of a solid in equilibrium with a gas at a pressure p; a certain portion of the surface will be covered by adsorbed atoms. Let the sites or adsorption centers be represented by a regular, two-dimensional array of

Figure 1. Part of a regular, infinite, two-dimensional, hexagonal array of adsorption sites. Full circles indicate sites covered by an adsorbed atom.

circles such as depicted in Figure 1. If we assume that any center can accommodate at most one atom, every site is either full (as indicated by the symbol ●) or empty (as indicated by the symbol ○). Given prescribed experimental conditions and assuming that equilibrium prevails, one wishes to determine (*a*) the fraction of filled sites, and (*b*) the arrangement of adsorbed particles among sites. As to (*b*), one can foresee several possibilities: at one extreme, all atoms on the surface are clustered and occupy a set of adjacent sites, leaving all others vacant (see Fig. 2a); at the other extreme, all atoms are evenly distributed among the sites (see Fig. 2b). Alternately, a random arrangement of adsorbed atoms (as represented by Fig. 1) and all intermediate degrees of order or disorder can be encountered. Furthermore, given a highly ordered (disordered) phase, one might inquire whether such a configuration is stable or whether transitions to a disordered (ordered) state are possible. These questions will be answered in the sections to follow. However, it is intuitively evident that the surface arrangement is significantly affected by interactions among the adsorbed atoms. If these are subject to strong attractive forces they tend toward the state of clustering symbolized by Figure 2a; if they repel one another the system will approach the distribution shown in Figure 2b. It will later be seen that other factors must also be taken into consideration; however, the above discussion suggests one problem that must be taken up in due time, namely the general question of interactions among a large number of particles held in fixed positions on a surface.

Figure 2. Extreme degrees of ordering of adsorbed atoms on the lattice of Figure 1.

The Lattice Representation

Having posed the problem, we divide the process of obtaining the solution into several stages. The first of these involves a simplification of the system under consideration, since it is obviously impracticable to deal with all possible interactions encountered over the entire lattice. For this purpose one introduces the *lattice representations;* these are collections of site groupings abstracted from the lattice. Several possible representations of the network of Figure 1 are shown in Figure 3. In simplest terms, the original lattice may be regarded as a collection of sites or *points* (Fig.

[1] Operated with support from the U. S. Army, Navy, and Air Force.

[2] Hijmans, J., and de Boer, J., *Physica*, 21, 471, 485, 499 (1955).

[3] Domb, C., *Advances in Phys.*, 9, 149 (1960).

3a). In the next degree of complexity Figure 1 is decomposed into a collection of points and *bonds* (Fig. 3b). Next in order of complexity is the occurrence of the triangle in the representation (Fig. 3c); if one wishes to obtain still better approximations, it is necessary to utilize the rhombus (Fig. 3d), the hexagon (Fig. 3e),

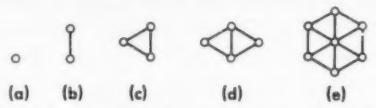

Figure 3. Lattice representations for the hexagonal array of Figure 1.

and higher geometric arrays. We shall refer to the most complicated pattern under consideration as the *basic figure* and to all others of lower order as *subfigures*. Subfigures appropriate to the square lattice array of Figure 4 are depicted in Figure 5. Anticipating somewhat, it is seen that once the lattice decomposition has been accomplished, the problem of considering lateral interactions among the adsorbed species has been tremendously reduced. Thus, if the lattice is considered simply as a collection of isolated points, no lateral interactions whatever can be taken into consideration.

Figure 4. Part of a regular, infinite, two-dimensional, square array of adsorption sites.

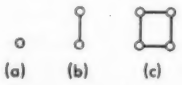

Figure 5. Lattice representations for the square array of Figure 4.

If the lattice is represented as a superposition of bonds and points, then nearest neighbor interactions can be taken into account. Using rhombi or squares as basic figures in the representation of hexagonal or square lattice arrays permits consideration of interactions between next nearest as well as nearest neighbors, and so on. Obviously, the price paid for these simplifications is a loss in accuracy of information gained from the theory. However, in going to successively larger basic figures one ought to be able to approximate the properties of the original lattice to any desired degree. In practice one is limited by the rapidly increasing complexity in mathematical analysis to the very simplest basic figures.

Having decided on the choice of the basic figure and of the subfigures, one must correctly tally the number of each type that is to be included in the representation. In the simplest case where the lattice representation consists of individual points there is no problem: if the lattice contains L centers, L points are required in the representation. The next higher approximation involving bonds and points is slightly more involved. Let Z represent the number of nearest neighbors to any given lattice site, i.e., its coordination number ($Z = 6$ in Fig. 1; $Z = 4$ in Fig. 4). Each site represents the intersection of Z straight connections to nearest neighbors; since a site shares each bond with one other point, the number of bonds per point is $Z/2$. Hence, the number of bonds for L sites is $ZL/2$, and this represents the number of bonds required in the lattice representation. One difficulty immediately becomes apparent; in the above-mentioned representation, each bond terminates in two points and the bond collection therefore already includes ZL points, whereas the correct number in the lattice is L. To rectify this overcount we introduce an additional assembly of $(1 - Z)L$ points. Then the collection of $LZ/2$ bonds and the additional $(1 - Z)L$ points correctly represents the lattice to this degree of approximation. The fact that $(1 - Z)L$ is negative need cause no undue concern: the situation arises because of the need for a corrective count; the total number of points in the collection of the basic figure and the subfigure is positive. The occurrence of negative numbers in the Hijmans-de Boer approach is part of the mathematical formalism required to arrive at self-consistent results.

Let us next consider the case where the triangle is used as a basic figure in the representation of Figure 1. Again, every site is an apex for Z adjacent triangles placed symmetrically about the lattice point under consideration. Since the point shares each triangle with two other points, the number of triangles per site is $Z/3$, and the number of triangles for L points is $LZ/3$. Thus, the lattice representation must contain $LZ/3$ triangles to start with. This assembly already includes LZ bonds (each triangle contains three sides), and it is therefore necessary to introduce an additional collection of $-ZL/2$ bond subfigures to obtain a correct total count of $ZL/2$ bonds in the lattice. The number of points contained in $ZL/3$ triangles and $-ZL/2$ points cancel out; hence a subfigure assembly of L points is needed to obtain a correct over-all tally. By this type of bookkeeping procedure one can decompose any lattice systematically into the proper number of simpler components.

Specification of Configurations

The second stage of the procedure consists in specifying the *configurations* associated with each type of subfigure. These are shown in Figure 6 for the lattice representations of the hexagonal network. Multiple representations of a given arrangement are not shown. For example, in Figure 6 the configuration T2 may be replicated by transferring the filled circle to the adjacent vertex point of the triangle. This type of multiplicity will be taken into account at a later stage. In the simplest case an isolated site is either empty or full; for pairs of sites the termini of a bond are either both empty, both filled, or one is empty and the other filled. For the triangular grouping the three apex points can be filled in succession. The more elaborate representations for the rhombus configurations illustrate the fact

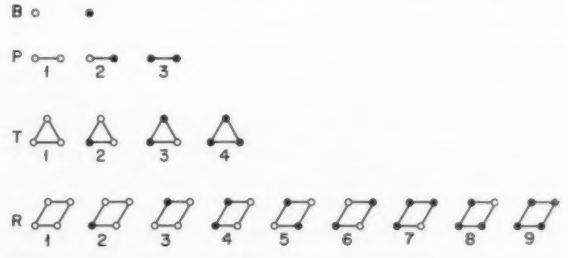

Figure 6. Various possible configurations pertaining to the lattice representations for the hexagonal array.

Table 1. Configurations for the Point Representation of a Lattice of L Sites

(1) Figure	(2) Designation of state	(3) Probability of occurrence	(4) Number of sites in state A or B	(5) Energy of configurations	(6) Energy of each set
○	A	α_0	$L\alpha_0$	ϵ_A	$L\alpha_0\epsilon_A$
●	B	α_1	$L\alpha_1$	ϵ_B	$L\alpha_1\epsilon_B$

Table 2. Configurations, Energies, and Probabilities for the Members of the Bond and Point Figure Assemblies

(1) Tally	(2) Figure	(3) Configuration sequence	(4) Probability of occurrence	(5) Energy parameter	(6) Total number of each set	(7) Total energy of each set	(8) Total number of *filled* sites in each configuration
$(1-Z)L$	○	A	α_0	ϵ_A	$(1-Z)L\alpha_0$	$(1-Z)L\alpha_0\epsilon_A$	None
	●	B	α_1	ϵ_B	$(1-Z)L\alpha_1$	$(1-Z)L\alpha_1\epsilon_B$	$(1-Z)L\alpha_1$
$\frac{ZL}{2}$	○—○	AA	β_0	ϵ_{AA}	$\frac{Z}{2}L\beta_0$	$\frac{Z}{2}L\beta_0\epsilon_{AA}$	None
	○—●	AB	$2\beta_1$	ϵ_{AB} ($\epsilon_{AB} \equiv \epsilon_{BA}$)	$\frac{Z}{2}L(2\beta_1)$	$\frac{Z}{2}L(2\beta_1)\epsilon_{AB}$	$ZL\beta_1$
	●—○	BA		ϵ_{BA}			
	●—●	BB	β_2	ϵ_{BB}	$\frac{Z}{2}L\beta_2$	$\frac{Z}{2}L\beta_2\epsilon_{BB}$	$ZL\beta_2$

Total number of ● sites in the Figure Assembly: $N_t = (1-Z)L\alpha_1 + ZL\beta_1 + ZL\beta_2$

that sites encountered in more complex subfigures may not be equivalent. By this we mean that it is not possible, for example, to superpose configuration R2 with R3 in such a manner that all empty and all filled sites coincide.

The specification of configurations is of the utmost importance in the further quantitative treatment, as will now be shown. Consider first the simplest approximation in which the lattice is represented as a collection of L isolated points. As shown in Table 1, we let A and B designate an unoccupied and occupied lattice site respectively, and we let α_0 and α_1 represent the probability that an individual site is in the state (also known as the configuration) designated by A and B respectively. Then the number of empty and filled sites is given as $L\alpha_0$ and $L\alpha_1$. In the lattice representation involving both bonds and points the situation is surveyed in columns (2), (3), and (6) of Table 2; the entries are defined by analogy to Table 1. It is noteworthy that bonds in the configuration AB are indistinguishable from those in the configuration BA, hence the probability of occurrence of each is β_1 and the probability of encountering both is $2\beta_1$. The total number of points and bonds in each of the possible distinguishable configurations is shown in Column (6) of Table 2.

Normalization and Consistency Relations

In the third step of the solution process we must take account of interrelations between α_0, α_1, β_0, β_1, and β_2; the latter quantities represent the unknowns in this problem. Two types of interrelations can be cited. The first arises from the requirement that the probability of encountering points in all possible configurations (A and B) or of encountering bonds in all possible configurations (AA, AB, BA, and BB) be unity. This is known as a *normalization condition*. We have, accordingly,

$$\alpha_0 + \alpha_1 = 1 \tag{1}$$

$$\beta_0 + 2\beta_1 + \beta_2 = 1 \tag{2}$$

The second set arises as follows: Suppose that the fraction of filled sites in the lattice proper is α_1, the number of filled lattice sites being $\alpha_1 L$. In the lattice representation consisting of bonds and points the number of filled sites is given by the sum of terms of Column (8) in Table 2. The two methods of description must match: hence, the requirement that

$$(1-Z)L\alpha_1 + ZL\beta_1 + ZL\beta_2 = L\alpha_1 \tag{3a}$$

which simplifies to

$$\alpha_1 = \beta_1 + \beta_2 \tag{3b}$$

An equation of the form (3) is known as a *consistency condition.*

Equilibrium Conditions Using the Quasichemical Approach

In the fourth stage of the procedure we set up conditions which specify the equilibrium state of the system. To keep the requisite mathematical techniques to an absolute minimum, we proceed by an analogy which has often been successfully employed in the literature: we regard the empty and full lattice sites as well as the particles in the gas phase as *bona fide* chemical species and we represent adsorption processes by chemical reactions. These will be considered as if they are subject to the laws of mass action; equilibrium constants are then written out in the usual manner. We shall verify in later sections that the procedure contemplated for adoption here leads to correct results.

As a simple example, consider the adsorption process on a lattice when use of the point as a basic figure in the lattice representation is justifiable. The process of filling an isolated site can now be expressed in terms of the "chemical reaction"

$$\mathrm{G} + \bigcirc \rightleftarrows \bullet \tag{4}$$

where G is an atom in the gas phase. The equilibrium constant for the above reaction is given by

$$K_0 = \frac{X_\bullet}{pX_\bigcirc} = \frac{(L\alpha_1/L)}{p(L\alpha_0/L)} \tag{5}$$

where p is the gas pressure and X the mole fraction of

empty or full sites. Equation (5) may be solved for

$$p = K_1\left(\frac{\alpha_1}{1-\alpha_1}\right) \equiv K_1\frac{\theta}{(1-\theta)} \tag{6a}$$

or

$$\alpha_1 \equiv \theta = \frac{p}{p + K_1} \tag{6b}$$

where Equation (1) and the definition $K_1 = 1/K_0$ has also been utilized. The above represents an isotherm equation; for, it specifies the fraction of filled sites, $\theta \equiv \alpha_1$, when the surface is in equilibrium with gas at a pressure p. Equation (6) is the well-known *Langmuir isotherm*, which is applicable providing all surface sites are considered equivalent and all interactions among adsorbed particles can be neglected.

A case of greater interest arises when it is desired to represent the lattice by bond and point figure assemblies. In this case, the adsorption of gas particles on a bond is written schematically as

$$2\mathrm{G} + \mathrm{O{-}O} \rightleftharpoons \bullet\text{—}\bullet \tag{7}$$

which can be thought of as a "net chemical reaction" for the two-step process $\mathrm{G} + \mathrm{O{-}O} \rightleftharpoons \mathrm{O{-}}\bullet$ and $\mathrm{G} + \mathrm{O{-}}\bullet \rightleftharpoons \bullet\text{—}\bullet$.[4]

The net reaction (7) must be accompanied by changes in occupation states of the point figure assembly via reaction (4), in order to prevent the type of overcounting mentioned in the first section. To see what is required, let us suppose that as a result of the reaction (7), Y bonds change their configuration from AA to BB and let us define a fraction f by $Y = L(Z/2)f$. Then reaction (7) is equivalent to the following sequence: (*a*) *add* $L(Z/2)f$ bonds in configuration BB *to* and (*b*) *remove* $L(Z/2)f$ bonds in configuration AA *from* the bond figure assembly. To keep pace with these processes it is necessary (*c*) to *add* $L(1-Z)f$ points in configuration B *to* and (*d*) to *subtract* $L(1-Z)f$ points in configuration A *from* the point figure assembly. Furthermore, corresponding to (*a*) and (*c*), we transfer (*e*) LZf atoms of type G and (*f*) $L(1-Z)f$ atoms of type G from the gas phase to the surface; by contrast, processes (*b*) and (*d*) do not require or release G atoms. In keeping with the customary chemical conventions, all species consumed in a process (or removed or caused to disappear from a system by the reaction) are written on the left; all species produced (or caused to be added to a system as the result of the reaction) are written on the right. Accordingly, the information carried in steps (*a*), (*b*), and (*e*) can be rewritten as the chemical equation

$$\{LZf\}\mathrm{G} + L(Z/2)f\,\mathrm{O{-}O} \rightleftharpoons L(Z/2)f\,\bullet\text{—}\bullet$$

Similarly, steps (*c*), (*d*), and (*f*) can be rewritten as

$$\{L(1-Z)f\}\mathrm{G} + L(1-Z)f\,\mathrm{O} \rightleftharpoons L(1-Z)f\,\bullet$$

The latter two equations can be added to yield a final "reaction" for the adsorption process on a lattice represented by bond and point figure assemblies:

$$\mathrm{G} + (Z/2)\,\mathrm{O{-}O} + (1-Z)\,\mathrm{O} \rightleftharpoons (Z/2)\,\bullet\text{—}\bullet + (1-Z)\,\bullet \tag{8}$$

with which we can associate the "equilibrium constant" K_2 in the expression

$$K_2 = \left(\frac{X_{\bullet\text{—}\bullet}^{Z/2}}{X_{\mathrm{O{-}O}}^{Z/2}}\right)\left(\frac{X_{\bullet}^{1-Z}}{X_{\mathrm{O}}^{1-Z}}\right)\left(\frac{1}{p}\right) \tag{9}$$

Or, writing $K_3 \equiv 1/K_2$,

$$p = K_3\left(\frac{\beta_2}{\beta_0}\right)^{(Z/2)}\left(\frac{\alpha_0}{\alpha_1}\right)^{(Z-1)} \tag{10}$$

which is one of two equilibrium relations which can be written down.

So far we have considered only the possibility of converting AA to BB configurations. It is evident that, having once created these units, one can generate the AB configuration by the "reaction"

$$\mathrm{O{-}O} + \bullet\text{—}\bullet \rightleftharpoons 2\,\mathrm{O{-}}\bullet \tag{11}$$

without occasioning any other changes; the number of occupied or unoccupied sites is not altered as a result of Equation (11), only the distribution of occupation states in the bond figure assembly is changed. We can characterize Equation (11) by another equilibrium constant C_1

$$C_1 = \frac{X_{\mathrm{O{-}}\bullet}^{2}}{X_{\mathrm{O{-}O}}\,X_{\bullet\text{—}\bullet}} = \frac{\beta_1^2}{\beta_0\beta_2} \tag{12a}$$

and this is the second equilibrium relation which was sought. It will be convenient to set $C \equiv 1/C_1$ and to write

$$\frac{\beta_0\beta_2}{\beta_1^2} = C \tag{12b}$$

We may solve Equation (12b) for $\beta_2/\beta_0 = C(\beta_1/\beta_0)^2$ and then substitute this result into Equation (10), which now reads

$$p = K(\beta_1/\beta_0)^Z\,(\alpha_0/\alpha_1)^{(Z-1)} \tag{13}$$

with $K = K_3C^{Z/2}$.

For later convenience we denote by *zero* and *first order cases* those mathematical relationships which apply when the lattice is represented by a point or by a superposition of bond and point figure assemblies, respectively.

A verification of Equations (12) and (13) is provided in Appendix A, where the results are obtained using statistical mechanics. At the same time, expressions are provided for the constants K and C appearing in Equation (12b) and (13). Since the specification of C will also be needed in later sections of the main text, we again resort here to a simple argument. It is well known that the equilibrium constant for any reaction is related to the standard free energy change by the expression $\Delta F^0 = kT\ln K$, where k is Boltzmann's constant and T the absolute temperature. As applied to reaction (11), the standard state for each species is most conveniently considered the entire bond figure assembly in the configuration AA, AB, or BB. When the entire assembly is in one particular state the configurational entropy vanishes. Since we have so far tacitly ignored the thermal entropy and energy, it follows from the above that $\Delta F^0 = \Delta E^0$, where ΔE^0

[4] The reason it suffices to consider the net reaction is that at this stage we are concerned only with the number of sites that are being occupied in the process of adsorption. For this purpose it is convenient to deal with the completely ordered state in which bonds are either completely filled (BB configuration) or completely empty (AA configuration). The redistribution of bond occupation states so as to introduce the AB configuration without incurring other changes will be accomplished as a subsequent step in the derivation.

is the change in energy accompanying reaction (11) when all reagents and products are held in their respective standard states. Thus, $-\Delta E_0^0/kT = \ln C_1$, where C_1 is the equilibrium constant for (11); moreover from column (5) of Table 2 one finds that

$$1/C_1 = C = e^{(2\epsilon_{AB} - \epsilon_{AA} - \epsilon_{BB})/kT} \quad (14)$$

or, in conjunction with Equation (12b)

$$\ln\beta_0 + \ln\beta_2 - 2\ln\beta_1 = (2\epsilon_{AB} - \epsilon_{AA} - \epsilon_{BB})/kT \quad (15)$$

This completes our task of presenting the relations applicable to the first order problem.

The Adsorption Isotherm for the First Order Case

The final stage in the solution of the first order problem consists in solving the set of simultaneous equations (1), (2), (3b), (12b), and (13), so that p may be determined as a function of α_1; this yields the desired isotherm equation. As it is not the object of this publication to discuss adsorption problems in detail, and since this stage is a problem in algebra, we will provide only a brief outline of the steps and of the results. Rewrite Equation (13) as

$$\beta_1/\beta_0 = Ra_1 \text{ where } R = (p/K\,a_1)^{1/Z} \text{ and } a_1 = \alpha_1/\alpha_0 \quad (16)$$

Then Eq (12b) reads

$$\beta_2/\beta_0 = C(Ra_1)^2 \quad (17)$$

Now from Equations (1)–(3) it follows that $\alpha_0 = \beta_0 + \beta_1$; hence

$$a_1 = \frac{\alpha_1}{\alpha_0} = \frac{\beta_1 + \beta_2}{\beta_0 + \beta_1} = \frac{\beta_1/\beta_0 + \beta_2/\beta_0}{1 + \beta_1/\beta_0} = \frac{Ra_1(1 + CRa_1)}{1 + Ra_1}$$

and this may be converted into a polynomial in R, namely

$$Ca_1R^2 + (1 - a_1)R - 1 = 0 \quad (18)$$

The latter has the solution

$$R = [a_1 - 1 \pm \sqrt{(1 - a_1)^2 + 4Ca_1}]/2Ca_1$$
$$= \{2a_1 - 1 \pm \sqrt{1 + 4a_1(1 - a_1)(C - 1)}\}/2Ca_1 \quad (19)$$

Here, use has been made of Equation (1) in arriving at the final formulation under the square root. We ignore the negative sign in (19) as being physically meaningless, and replace the square root with the symbol

$$B = \sqrt{1 + 4a_1\,(1 - a_1)(C - 1)}.$$

In view of the definitions for B, K and C, we obtain from Equation (19) the final relation

$$\frac{p}{K} = \frac{\theta}{1 - \theta}\left(\frac{B - 1 + 2\theta}{2\theta}\right)^Z e^{Zw/kT} \quad (20)$$

where, in accord with usual practice, we have set $\alpha_1 \equiv \theta$. Equation (20) is known as the *Fowler-Guggenheim isotherm equation*. Equation (20) with $C = 2.5$ or $C = 3$ and $Z = 6$ is plotted in Figure 7; the case $C = 1$ reduces to the Langmuir isotherm, equation (6), which is also plotted. The presence of loops (dotted in Fig. 7) in the isotherms indicates the onset of two-dimensional condensation; the system spontaneously transforms from a "composition" denoted by θ_1 to that denoted by θ_2 as the pressure is raised infinitesimally beyond the value p_c.

We note in passing that the effect of next nearest neighbors cannot be taken into account until the rhombus of Figure 3D is selected as the basic figure. The appropriate theory for the case has recently been published.[5]

The Energy, Entropy, and Free Energy of the Surface Phase

Before proceeding to the next topic it is necessary to obtain expressions for the energy E, entropy S, and free energy F, of particles occupying surface sites. Consider first the zero order case and designate the energies of an empty and filled lattice point as ϵ_A and ϵ_B, respectively. Although the zero of energy is arbitrary and one can set $\epsilon_A = 0$, for greater generality the present scheme will be retained. As before, let α_0 and α_1 represent the probability that an individual site is empty or filled. Referring to Table 1 we see that the energy of the filled and empty sites taken together is

$$E = L\{\alpha_0\epsilon_A + \alpha_1\epsilon_B\} \quad (21)$$

Furthermore, the probabilities are connected to the entropy of the system by the familiar relation

$$S = k\ln\Omega = k\ln\{L!/(L\alpha_0)!(L\alpha_1)!\} \quad (22)$$

where k is Boltzmann's constant and Ω is the number of ways of arranging the L sites such that $L\alpha_0$ are empty and $L\alpha_1$ are full. Since L is a large quantity we can employ Stirling's approximation for large n

$$\ln(n!) \cong n\ln n - n$$

to rewrite (22) as

$$S = k\{L\ln L - L - L\alpha_0\ln L\alpha_0 + L\alpha_0 - L\alpha_1\ln L\alpha_1 + L\alpha_1\} \quad (23)$$

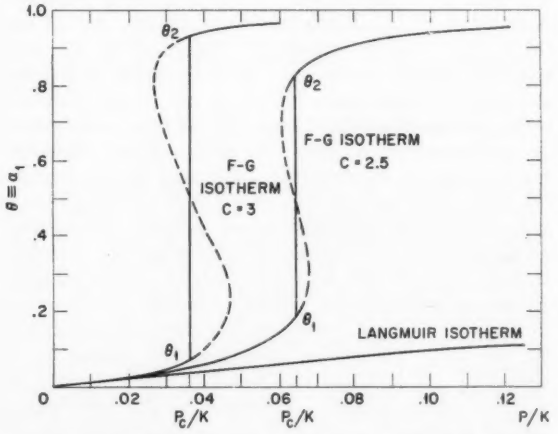

Figure 7. Typical isotherm equations computed according to Equations (6b) and (20). In the latter $Z = 6$; P_c is the pressure where two-dimensional condensation occurs.

The nonlogarithmic terms cancel on account of Equation (1). If we rewrite terms of the form $L\alpha_1\ln L\alpha_1$ as $L\alpha_1\ln L + L\alpha_1\ln\alpha_1$, mutual cancellations again occur on account of Equation (1) for all terms involving $\ln L$. This leaves the simple expression

$$S = -kL\{\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1\} \quad (24)$$

where $S > 0$ because $0 < \alpha_1 < 1$.

One can now immediately construct the expression for the Helmholtz free energy:

[5] BUMBLE, S., AND HONIG, J M., *J. Chem. Phys.* **33**, 424 (1960).

$$F = E - TS = L[\alpha_0\epsilon_A + \alpha_1\epsilon_B] + LkT\,[\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1] \quad (25)$$

which is needed for later reference.

Reviewing our progress to this point we see that, starting (*a*) with the probability variables α_0 and α_1 and (*b*) with the energy parameters ϵ_A and ϵ_B, we have set down simple expressions for the energy, entropy, and free energy of the representation for the zero order case.

Let us now repeat these steps for the lattice representation consisting of the bond and the point. The situation is somewhat more involved, but the basic procedure remains the same. Table 2 provides a survey of the configurations encountered in the figure assemblies, of their probabilities, and of their energies. The quantities ϵ_{AA}, ϵ_{AB}, ϵ_{BB} are defined by analogy to Table 1.

By a simple extension of Equation (21), the energy of the lattice representation is found to be

$$E = (1 - Z)\,L\{\alpha_0\epsilon_A + \alpha_1\epsilon_B\} + (ZL/2)\,\{\beta_0\epsilon_{AA} + 2\beta_1\epsilon_{AB} + \beta_2\epsilon_{BB}\} \quad (26)$$

and in a fairly obvious extension of the procedure leading to Equation (24), one arrives at Equation (29). Readers wishing to verify these results are referred to the intervening steps presented below. The entropy expression reads

$$S = k\ln\Omega = k\ln\left\{\frac{(ZL/2)!}{(ZL\beta_0/2)![(ZL\beta_1/2)!]^2(ZL\beta_2/2)!} \times \frac{[(1 - Z)L]!}{[(1 - Z)L\alpha_1]!\,[(1 - Z)L\alpha_0]!}\right\} \quad (27)$$

In Equation (27), the first factor in the logarithmic argument refers to the number of ways that bonds in the configurations [column (3) of Table 2] AA, AB, BA, and BB, for the respective numbers shown in column (6), can be distributed among $ZL/2$ available choices. A similar factor obtains for the number of ways of distributing points in configuration A and B among the $(1 - Z)L$ sites of the point figure assembly. On applying Stirling's theorem to (27), one obtains

$$\begin{aligned} S \cong k\{&(ZL/2)\ln(ZL/2) - ZL/2 - (ZL\beta_0/2)\ln(ZL\beta_0/2) \\ &+ ZL\beta_0/2 - 2(ZL\beta_1/2)\ln(ZL\beta_1/2) + 2ZL\beta_1/2 \\ &- ZL\beta_2/2\ln(ZL\beta_2/2) + ZL\beta_2/2 \\ &+ (1 - Z)L\ln(1 - Z)L - (1 - Z)L \\ &- (1 - Z)L\alpha_1\ln[(1 - Z)L\alpha_1] + (1 - Z)L\alpha_1 \\ &- (1 - Z)L\alpha_0\ln[(1 - Z)L\alpha_0] + (1 - Z)L\alpha_0\} \end{aligned} \quad (28)$$

Considerable simplification is again achieved by use of the normalizing conditions, Equations (1) and (2). All nonlogarithmic terms cancel mutually; furthermore, if we write terms like $\ln(LZ\beta_0/2)$ or $\ln L(1 - Z)\alpha_0$ as $\ln(LZ/2) + \ln\beta_0$ or $\ln L(1 - Z) + \ln\alpha_0$, additional cancellations occur and we are left with the final expression

$$S = -kL(Z/2)[\beta_0\ln\beta_0 + 2\beta_1\ln\beta_1 + \beta_2\ln\beta_2] - kL(1 - Z)[\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1] \quad (29)$$

From (26) and (29) it is easy to construct the function $F = E - TS$, which is needed in later discussions. The important assumption utilized henceforth is that E, S, and F, as obtained by a consideration of figure assemblies, represent good approximations to the corresponding functions for the lattice proper.

Adaptation of the Theory to Binary Mixtures

The theory outlined above can readily be adapted to cover the statistical properties of solutions. One need merely recognize that the "empty site" may be considered as component A and the "filled site" as component B of a binary mixture. Insofar as the lattice model represents a reasonable approximation to liquids the development of the previous sections also applies to that category of binary mixtures. One must now, however, deal with three-dimensional arrays of lattice sites, and this ordinarily would require three-dimensional figures for the lattice representations. Actually, this extension is not considered here as we shall henceforth restrict ourselves to the point and the bond representations. For these cases, passage from two to three dimensions involves only a change in the value of the coordination number Z.

Before we proceed further, it must be recognized that the composition of a solid or liquid solution is subject to adjustment by the experimentalist who adds appropriate amounts of the components to the mixture. By contrast, for a given adsorbent, the amount of adsorbed material in equilibrium with its vapor at a fixed pressure p is not arbitrarily adjustable. Mathematically, this is reflected in the fact that up to now α_1 has been regarded as an unknown quantity, to be computed once p, K, C and T have been specified. In applying order-disorder theory to binary solutions, however, α_1 is to be regarded as an adjustable parameter, not as an unknown.

Another point to be kept in mind is that L has so far been considered to be constant. Clearly, this was appropriate to the gas adsorption problem, but is no longer pertinent to binary mixtures, where the composition can be altered by further addition of either component. As more material is added the number of lattice sites is increased. The consequences of these changes will emerge shortly.

The Zero Order Approximation as Applied to Binary Liquid Mixtures

We consider now the statistical properties of a liquid mixture in which the lattice is represented by the zero-order approximation. In this case Equations (21) and (24) are applicable. Writing out $N_A = L\alpha_0$, $N_B = L\alpha_1$ and the mole fraction $\alpha_i = N_i/(N_A + N_B)$ where i = A, B we obtain for the free energy of the mixture

$$F = N_A\epsilon_A + N_B\epsilon_B + kT\{N_A\ln[N_A/(N_A + N_B)] + N_B\ln[N_B/(N_A + N_B)]\} \quad (30)$$

Since the first two terms on the right can be considered as the energies of an isolated assembly of $N_A(N_B)$ units of component A(B), the free energy of mixing is given by

$$F_m = kT\left\{N_A\ln\frac{N_A}{N_A + N_B} + N_B\ln\frac{N_B}{N_A + N_B}\right\} \quad (31)$$

In the zero order case no interaction between molecules in the mixture is taken into account, hence $E_m = 0$ and the entropy of mixing is found from Equation (31) as $S_m = -F_m/T$. Solutions subject to these relations are said to be *ideal*.

The chemical potential of component A may be computed from Equation (30) by differentiation with respect to N_A at fixed T and N_B; it is to be noted that L is not considered constant here and that N_A can be changed independently of N_B (in gas adsorption processes the constancy of L implies that for every site which is filled an empty site "disappears" from the

system, so that N_A and N_B are not independent of each other). Accordingly,

$$\mu_A = (\partial F/\partial N_A)_{T,N_B} = \epsilon_A + kT \ln [N_A/(N_A + N_B)] \quad (32)$$

The chemical potential per molecule of the pure component μ_A^0 is found from (32) by setting $N_B = 0$; thus $\mu_A^0 = \epsilon_A$ and

$$\mu_A - \mu_A^0 = kT \ln[N_A/(N_A + N_B)] \quad (33)$$

The chemical potential per mole $\tilde{\mu}_A$ is given by $\tilde{\mu}_A = \tilde{\mu}_A^0 + RT \ln [M_A/(M_A + M_B)] = \tilde{\mu}_A^0 + RT\ln\alpha_0$ where M_A and M_B denote the mole numbers of the two components and α_0 is the mole fraction of A. At equilibrium the chemical potential of component A in the gas phase, $\tilde{\mu}_A^{(G)} = \tilde{\mu}_A^{0(G)} + RT\ln p_A$, equals that of A in the solution. Hence

$$\tilde{\mu}_A^{0(G)} + RT \ln p_A = \tilde{\mu}_A^0 + RT \ln\alpha_0 \quad (34)$$

But for $\alpha_0 = 1$ (i.e., when the "solution" consists of pure A), p_A represents the vapor pressure p_A^0 of the pure component; it follows from (34) that $RT\ln p_A^0 = \tilde{\mu}_A^0 - \tilde{\mu}_A^{0(G)}$, whence we find that

$$p_A/p_A^0 = \alpha_0 \quad (35)$$

which is an expression of *Raoult's law*. A similar formula $p_B/p_B^0 = \alpha_1$ obtains for component B. The above derivation indicates that some very fundamental properties of solutions may be derived as special cases of order-disorder theory.

The First Order Approximation as Applied to Binary Mixtures

We now repeat the derivations in the last section on a slightly more sophisticated level, by allowing for interactions between nearest neighbors in the binary solution. For this purpose we resort to the first order scheme, wherein the "lattice" of the liquid is represented by the bond as the basic figure and the point as the subfigure. Again, the "empty site" and "full site" are now identified with component A and B, respectively. Equations (1), (2), and (3) can be taken over without change, and Equations (26) and (29) now apply. However, since α_1 is an adjustable parameter, only the "chemical reaction" (11) is relevant. [Rearrangements in conformity with (11) do not affect the values of N_A or N_B.] Thus, Equation (10) must be ignored while Equation (12) can be adopted as it stands.

Our immediate objective now is to obtain the function $F(N_A, N_B, T)$ corresponding to Equation (30). For this purpose it seems simplest to substitute $\alpha_1 - \beta_1$ for β_2 and $\alpha_0 - \beta_1$ for β_0 wherever β_2 and β_0 occur in Equation (26) and as coefficients of logarithmic terms in (29). [The equality $\alpha_0 = \beta_0 + \beta_1$ can be deduced from Equations (1), (2), and (3).] This leads to the expression

$$\begin{aligned} E - TS = F = (Z/2)L[(\alpha_0 - \beta_1)\epsilon_{AA} + 2\beta_1\epsilon_{AB} + \\ (\alpha_1 - \beta_1)\epsilon_{BB}] + (1 - Z)L[\alpha_0\epsilon_A + \alpha_1\epsilon_B] + \\ kT[(Z/2)L\{(\alpha_0 - \beta_1)\ln\beta_0 + 2\beta_1\ln\beta_1 + \\ (\alpha_1 - \beta_1)\ln\beta_2\} + (1 - Z)L(\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1)] \end{aligned} \quad (36)$$

If we introduce Equation (15) into (36) we obtain

$$\begin{aligned} F = (Z/2)L(\alpha_0\epsilon_{AA} + \alpha_1\epsilon_{BB}) + (1 - Z)L(\alpha_0\epsilon_A + \alpha_1\epsilon_B) + \\ kT[(Z/2)L\{\alpha_0\ln(\alpha_0 - \beta_1) + \alpha_1\ln(\alpha_1 - \beta_1)\} + \\ (1 - Z)L\{\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1\}] \end{aligned} \quad (37)$$

The above equation serves as the starting point for the calculation of thermodynamic properties of binary solutions; unfortunately, the requisite mathematical manipulations turn out to be very unwieldy. Let us therefore introduce simplifications to minimize the mathematical labor without destroying the main features of first order theory. For this purpose we shall assume that the quantity w defined by

$$w = \epsilon_{BB} + \epsilon_{AA} - 2\epsilon_{AB} = -kT\ln C \quad (38)$$

is small compared to unity. It is shown in Appendix B that for $w/kT \ll 1$, Equation (37) reduces to

$$\begin{aligned} F = (Z/2)L(\alpha_0\epsilon_{AA} + \alpha_1\epsilon_{BB}) + (1 - Z)L(\alpha_0\epsilon_A + \alpha_1\epsilon_B) + \\ kTL\{\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1 - \alpha_0\alpha_1 Zw/2kT\} \end{aligned} \quad (39)$$

Now set $\chi_A = (Z/2)(\epsilon_{AA} - 2\epsilon_B)$, $\chi_B = (Z/2)(\epsilon_{BB} - 2\epsilon_B)$ and substitute for α_0 and α_1 in terms of N_A and N_B. Then

$$\begin{aligned} F = N_A\epsilon_A + N_B\epsilon_B + N_A\chi_A + N_B\chi_B + \\ kT\left\{N_A\ln\frac{N_A}{N_A + N_B} + N_B\ln\frac{N_B}{N_A + N_B}\right\} - \frac{N_A N_B}{N_A + N_B}\frac{Zw}{2} \end{aligned} \quad (40)$$

which is the final desired expression.

From this point on we closely follow the discussion of the preceding section. The chemical potential of the solution is obtained from (40) as

$$\begin{aligned} \mu_A(N_A, N_B, T) = \left(\frac{\partial F}{\partial N_A}\right)_{T,N_B} = \mu_A^0 + kT\ln\left(\frac{N_A}{N_A + N_B}\right) - \\ \left(\frac{N_B}{N_A + N_B}\right)^2\frac{Zw}{2} \\ = \mu_A^0 + kT\ln\alpha_0 - \alpha_1^2(Zw/2) \end{aligned} \quad (41)$$

where $\mu_A^0 = \mu_A(N_A, O, T)$. It is evident from comparison with the standard thermodynamic relation $\tilde{\mu}_A = \tilde{\mu}_A^0 + RT\ln x_A + RT\ln\gamma_A$ that the activity coefficient γ_A for the solution is given by

$$\ln\gamma_A = -(1 - \alpha_0)^2 Zw/kT \quad (42)$$

and that

$$\mu_A^0 = \epsilon_A + \chi_A \quad (43)$$

so that by means of our present formalism we have been led to an explicit relation for the activity coefficient and for μ^0; these quantities always remain unspecified in a purely thermodynamic treatment. By following the procedure for adaptation of the theory to binary mixtures, we obtain Raoult's law in the form

$$p_A = p_A^0\alpha_0 e^{-\alpha_1^2 Zw/2kT} \quad (44)$$

The free energy of mixing is given by

$$F_m = kTL\{\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1\} - L\alpha_0\alpha_1 Zw/2 \quad (45)$$

The first set of terms in (45) is identical with (31); hence the entropy of mixing is represented by the (positive) quantity

$$S_m = -kL\{\alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1\} \quad (46)$$

whereas the energy of mixing is given by

$$E_m = -L\alpha_0\alpha_1 Zw/2 \quad (47)$$

Mixtures satisfying the above-mentioned criteria are said to be *strictly regular*. For $w = 0$, the solutions are ideal.

It will be noted that as w/kT deviates from unity the solutions become progressively less ideal. For sufficiently negative values of w/kT [i.e., for conditions

where ϵ_{AB} is greater than $(\epsilon_{AA} + \epsilon_{BB})/2$ and at sufficiently low temperature] a very interesting phenomenon takes place, which is most easily understood by reference to Figure 8, where p_A/p_A^0 of Equation (44) is schematically plotted against the mole fraction α_0 of component A. For $-(Zw/2kT) < 2$ a curve of type 1 results, but for $-(Zw/2kT) > 2$ a curve of type 3 is obtained. Under the latter circumstance a mixture of components for which α_0 lies between the limits L and M will split into two phases having the compositions indicated by L and M, respectively. The crude statistical analysis presented here as a very special case of order-disorder theory is thus able to predict the phenomenon of critical mixing. The condition at which this phenomenon first sets in is given by the conditions $\partial F_m/\partial\alpha_1 = \partial^2 F_m/\partial\alpha_1^2 = 0$, which in view of Equations (1) and (45) lead to the mathematical requirements

$$\ln[\alpha_1/(1 - \alpha_1)] = -(2\alpha_1 - 1)Zw/2kT \qquad (w < 0) \quad (48)$$

and

$$\alpha_1(1 - \alpha_1) = -kT_c/Zw \qquad (w < 0) \quad (49)$$

Equation (48) is satisfied for $\alpha_1 = {}^1/_2$ as may be verified by direct substitution, and with this value of α_1, the temperature of critical mixing is seen to be

$$T_c = -Zw/4k \qquad (w < 0) \quad (50)$$

A more extensive discussion of critical phenomena is provided at the conclusion of the next section.

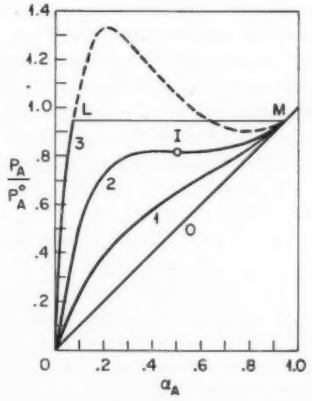

Figure 8. Ratio of partial vapor pressures of regular solutions. P_A/P_A° is computed according to Equation (44) for the various $(-Zw/2kT)$ values entered alongside the curve. The straight line represents Raoult's law; as $-w$ increases ($w < 0$), departures from Raoult's law occur; a critical phenomenon sets in for $-Zw/2kT = 2$ at the inflection point marked *I*. The dotted curve marks the unstable region where the liquid splits into two phases for which the vapor pressure of each constituent has the same value in both phases. Adapted from Guggenheim.[6]

The Concept of Long-Range Order: Superlattices

In the last two sections the presentation has referred mainly to liquid mixtures under the assumption that the lattice model furnishes an adequate representation of a liquid. This presupposition is subject to debate, but there can be little doubt concerning the utility of the lattice model in the case of a binary solid mixture, particularly if the two components are of roughly equal size and if the role of lattice imperfections can be neglected.

In adapting order-disorder theory to the present situation we consider as a specific example the CuZn alloy (β brass). The atoms are arranged on a body-centered cubic lattice which may be depicted as a combination of two interpenetrating simple cubic lattices, as shown in Figure 9. The low-temperature arrangement is one in which all Cu atoms are located on one sublattice (consisting of a sites) and all Zn atoms on the other (consisting of b sites). At sufficiently high temperatures the two constituents are randomly distributed. We are interested in the process of rearrangement whereby the highly disordered, high temperature system is converted into the ordered, low temperature phase.

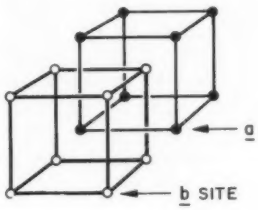

Figure 9. The ordered CuZn structure, as represented by two interpenetrating simple cubic lattices.

Consider a bcc lattice of L sites of which $L/2$ are occupied by Cu atoms (Type A constituent) and the remaining $L/2$ by Zn atoms (type B constituent). Let $Lr/2$ represent the number of A atoms on a sites and $L(1 - r)/2$ represent the number of A atoms on b sites. Then the number of B atoms on b and a sites is given by $Lr/2$ and $L(1 - r)/2$ respectively. The completely random arrangement is then characterized by the value $r = {}^1/_2$, because there is equal likelihood for finding an A atom on an a or b type site. The completely ordered arrangement is characterized by the value $r = 0$ or $r = 1$; because of the equivalence of the two sublattices, states characterized by r values diminishing from $r = {}^1/_2$ to $r = 0$ merely duplicate those specified by r values which increase from $r = {}^1/_2$ to $r = 1$. Hence it suffices to restrict r values to the range ${}^1/_2 \leq r \leq 1$. The distribution of A and B atoms among sites of type a and b for a given value of r is summarized in Table 3. In order to take interactions between nearest neighbors into account one must consider the population of immediately adjacent lattice sites. This distribution is also shown in Table 3. In the interpenetrating lattice of Figure 9, atoms forming nearest neighbor pairs belong to each of the two types of sublattices; for, the a-a distance between adjacent sites on the a sublattice (or between b-b sites

Table 3. Specification of the Occupation of the Lattice by A and B Atoms

Entry	Type of site	Type of atom	Probability of occurrence
1	a	A	$({}^1/_2)r$
2	a	B	$({}^1/_2)(1 - r)$
3	b	A	$({}^1/_2)(1 - r)$
4	b	B	$({}^1/_2)r$
5	$a - b$	A − A	$({}^1/_2)^2r(1 - r)$
6	$a - b$	A − B	$({}^1/_2)^2r^2$
7	$a - b$	B − A	$({}^1/_2)^2(1 - r)^2$
8	$a - b$	B − B	$({}^1/_2)^2r(1 - r)$

[6] Guggenheim, E. A., "Mixtures," Clarendon Press, Oxford 1952.

on the b sublattice) is greater than that between adjacent a and b sites. Hence only the a-b site arrangement is listed in entries 5–8. The arrangement is considered ordered if nearest neighbor a-b sites are in the configuration A-B or B-A; the configurations A-A or B-B are disordered.

The items in Table 3 can be arranged to read as follows: Entries 1 and 4 can be considered as right (R) and entries 2 and 3 as wrong (W) configurations. Similarly, entry 6 can be considered an R—R configuration, entry 7 as a W-W configuration, and the two remaining ones as R-W and W-R configurations. On this new basis the R entry is associated with the probability variable r, and the W entry with $(1 - r)$. The RR, WW and RW probabilities are designated by the variables σ_j in the manner indicated for the revised Occupation Specification, Table 4. Examination of this table shows that it is analogous to Table 2. Thus, we have succeeded in recasting the original problem in terms amenable to application of the order-disorder formalism. One item of specialization should be carefully noted: The quantities σ_j, analogous to β_j, are given by $\sigma_2 = \rho_1^2$, $\sigma_1 = 2\rho_1\rho_0$, $\sigma_0 = \rho_0^2$, where the ρ_j are equal to the α_j. These are precisely the interrelations between the β and α probabilities derived in Equations (80)–(82) listed in Appendix B as a special case of the first order approximation.

Table 4. Revised Tabulation of Lattice Occupation

Entry of Table 3	Configuration	Probability original		Variables new	Energy
1 + 4	R	r	$\equiv$	ρ_1	ϵ_R
2 + 3	W	$1 - r$	$\equiv$	ρ_0	ϵ_W
6	R − R	r^2	=	σ_2	ϵ_{RR}
7	W − W	$(1 - r)^2$	=	σ_0	ϵ_{WW}
5 + 8	R − W, W − R	$2(1 - r)r$	=	$2\sigma_1$	ϵ_{RW}

NOTE: The probability variables in r for the last three entries in this table were computed from the first two entries.

These plausibility arguments indicate that the results cited in Equations (39)–(50) apply to the present situation. The state of order is that in which the W-R configuration is nonexistent. Returning to Equations (12b) and (38) it is seen that σ_1 can be minimized by making w a large negative quantity, i.e., by requiring that ϵ_{AB} greatly exceed the quantity $(\epsilon_{AA} + \epsilon_{BB})/2$, or that the interaction energy between unlike atoms be much stronger than the average between like atoms.

At this point it becomes convenient to define the *degree of order* s by

$$r = \tfrac{1}{2}(1 + s) \quad \text{or} \quad s = 2r - 1 \qquad (51)$$

where the ordered state is represented by $s = 1$ and the disordered state by $s = 0$.

Using this definition, Equation (45) becomes

$$F_m = (kTL/2)\{(1 - s)\ln[(1 - s)/2] + (1 + s)\ln[(1 + s)/2]\} - L(1 - s^2)Zw/8 \qquad (52)$$

and Equation (48) for the values at which F_m becomes a minimum reads

$$\ln[(1 + s)/(1 - s)] = -sZw/2kT = 2sT_c/T \qquad (53)$$

with T_c defined by Equation (50). On the basis of (52) it is possible to gain a better insight on the phenomenon of order-disorder transitions. A plot of F_m/LkT versus s is shown in Figure 10 for different values

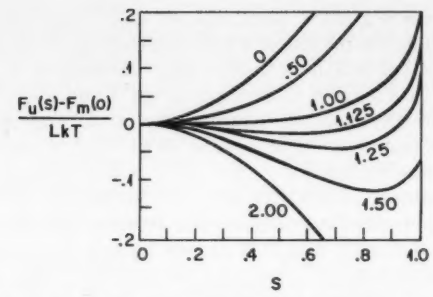

Figure 10. Plot of the free energy of mixing vs the degree of ordering for a variey of $-Zw/4kT = T_c/T$ values ($w < 0$, $T_c > 0$). $F_m(s)$ is the function (52); $F_m(0)$ is Equation (52) with $s = 0$. Adapted from Guggenheim.[6]

of $|Zw/4kT| = T_c/T$, when $w < 0$. We see that for small T_c/T ratios the lowest value of F_m occurs at $s = 0$; this solution satisfies (53) identically. However, as T_c/T is increased beyond unity, the minimum of F_m no longer occurs at $s = 0$ but shifts to positive s values. From the foregoing it is seen that for $w < 0$, the disordered state is the one corresponding to the lowest free energy of mixing as long as $T > T_c$. However, when $T < T_c$, the minimum occurs at successively larger s values and as $T \to 0$, $s \to 1$. This means that below a critical temperature the system can lower its total free energy by the process of ordering (in the technical sense defined earlier); therefore, the transition from disorder to order represents a spontaneous process which should be observable if kinetically feasible. The degree of order consistent with the minimum F_m at any given temperature $T < T_c$ may be determined by numerical solution of Equation (53); a plot of s versus T/T_c obtained in this manner is depicted in Figure 11. It is seen that the degree of ordering is rather perfect in the range $0 < T < T_c/2$, but that s diminishes sharply with increasing T beyond this range.

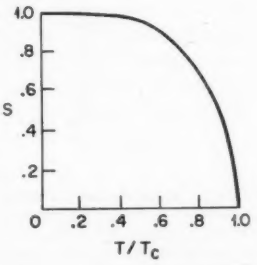

Figure 11. The degree of order s as a function of the ratio T/T_c in the range where order-disorder transitions may occur; s is a root of Equation (53).

Transitions of the type just discussed are known as *second order* transitions, and the critical temperature T_c is termed the *lambda point*, for reasons which will be clarified below. The question now remains as to how such a change in state can be experimentally determined. To deal with this problem, we note that Equation (47) for the energy of mixing may be rewritten as

$$E_m = -L(1 - s^2)Zw/8 \qquad (54)$$

The equilibrium value of s is that satisfying Equation (53), i.e., the value which minimizes F_m. Using either Equation (53) or Figure 11, the relation $E_m(s)$, Equation (54), may be converted into the function $E_m(T/T_c)$

which is depicted in Figure 12(a), where $E_m/L[Zw/8]$ is plotted against T/T_c. One observes that E_m remains essentially at zero until the value $T \simeq 0.3\ T_c$ is reached; E_m then increases with T and reaches its saturation value $-LZw/8$ ($w < 0$) at $T = T_c$. Using Figure 12(a) one can then calculate the heat capacity

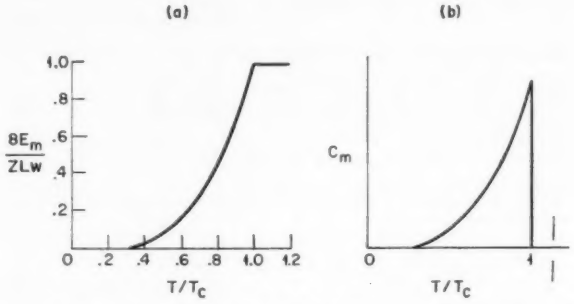

Figure 12. (a) The variation of E_m with T/T_c computed according to Equation (54), where s is a root of Equation (53). Adapted from Guggenheim.[6] (b) Schematic diagram of the configurational heat capacity of mixing vs T/T_c, obtained from Figure 12(a).

for mixing, C_m, as a function of temperature. This quantity, shown schematically in Fig. 12(b), exhibits a sharp discontinuity as the temperature passes through the critical value T_c. The shape of this curve is in the form of a Greek capital lambda, whence the name lambda point. Thus, transitions of the second order may be detected experimentally by carrying out heat capacity measurements as a function of T and searching for Λ-type discontinuities. Although the entire discussion is based on a very crude model, there are many systems in which this theory furnishes a fairly adequate description of the experimental findings. It will be noted that the value of the critical temperature varies linearly with w. When $w > 0$ (when the average of the interactions between like atoms exceeds that between unlike atoms) T_c formally assumes a negative value, i.e., there exists no second order phase transition.

It should be noted again that we have considered here the special case of a 50-50 composition alloy. The situation is more complicated for alloys in which the mole ratio of the constituents assumes other values.

Ferromagnetism

We conclude with a very brief discussion of ferromagnetism. Here a net magnetic moment m associated with each atom is aligned with or against an external magnetic field H. In addition, there exists an exchange interaction energy between nearest neighbors, denoted by $-J/2$ if the moments are parallel and by $+J/2$ if they are not. We now let configuration A represent a magnetic moment aligned with, and B an alignment against, the external field. Then, in the first order lattice representation the internal energy of the bond and point assemblies, exclusive of the interaction with the external field, is given by

$$E_1 = (ZL/2)[\beta_0(-J/2 + \epsilon_{AA}) + 2\beta_1(J/2 + \epsilon_{AB}) + \beta_2(-J/2 + \epsilon_{BB})] + (1 - Z)[\alpha_0\epsilon_A + \alpha_1\epsilon_B] \quad (55)$$

and the magnetic field interaction is

$$E_H = -LmH\{(Z/2)(2\beta_0 - 2\beta_2) + (1 - Z)(\alpha_0 - \alpha_1)\} = -LmH(\alpha_0 - \alpha_1) \quad (56)$$

where the right hand side was obtained from use of Equation (3b) and its counterpart involving α_0. The net interaction energy now reads

$$E = (Z/2)L\{(J/2)(2\beta_1 - \beta_0 - \beta_2) + \beta_0\epsilon_{AA} + 2\beta_1\epsilon_{AB} + \beta_2\epsilon_{BB}\} + (1 - Z)L\{\alpha_0\epsilon_A + \alpha_1\epsilon_B\} - LmH(\alpha_0 - \alpha_1) \quad (57)$$

The entropy of the system is still representable by Equation (29), and the consistency and normalization conditions apply as before. The remainder of the procedure then follows the method outlined in the section on equilibrium conditions and Appendix A. One thus arrives at the equilibrium relations

$$\beta_0\beta_2/\beta_1^2 = e^{2J - w/kT} \quad (58)$$

and

$$(\alpha_1/\alpha_0)^{Z-1}(\beta_0/\beta_2)^{Z/2} = e^{2mH/kT} \quad (59)$$

respectively. It will be noted that in these manipulations α_1 is considered as a variable rather than a parameter since the number of spins aligning with or against a fixed external field is not fixed by the experimentalist except indirectly through the magnitude of H.

It is not the object here to enter into a lengthy description of the phenomenon of ferromagnetism. We have just shown above that these properties again can be treated in terms of order-disorder concepts. Beyond this point it becomes necessary to solve Equations (58) and (59) together with the normalization and consistency conditions for each of the variables and to compute physical quantities of interest such as the Curie temperature, the spontaneous magnetization per atom, and the like. Readers interested in these matters are referred to the literature on this subject.[2,3]

Appendix A; Statistical Derivation of the Equilibrium Conditions and Evaluation of Equilibrium Constants

The aim of this section is to rederive Equations (12) and (13) and to obtain expressions for K and C. Reference to (26) and (29) shows that $F = E - TS$ is nominally a function of five variables, but due to the existence of the constraints (1), (2), and (3), only two variables are independent. It turns out to be convenient to select α_1 and β_2 as independent; we can then solve Equations (1)–(3) for α_0, β_0, and β_1 in terms of α_1 and β_2 as follows:

$$\alpha_0 = 1 - \alpha_1 \quad (60)$$

$$\beta_1 = \alpha_1 - \beta_2 \quad (61)$$

$$\beta_0 = 1 - 2\alpha_1 + \beta_2 \quad (62)$$

Since by choice $F = F(\alpha_1,\beta_2)$ we obtain two equilibrium conditions: The first states that

$$\mu^{(G)} = \mu^S = (1/L)[\partial F(\alpha_1,\beta_2)/\partial\alpha_1]_{\beta_2,T,L} \quad (63)$$

where $\mu^{(G)}$ is the chemical potential of the gaseous species which at equilibrium is the same as the chemical potential for the adsorbed phase. The second relation is a minimization condition

$$0 = [\partial F(\alpha_1,\beta_2)/\partial\beta_2]_{\alpha_1,T,L} \quad (64)$$

Taking (26) and (29) in conjunction we can now rewrite the equilibrium conditions as follows:

$$\mu^{(G)} = \frac{\partial(E - TS)}{\partial\alpha_1} + \left(\frac{\partial(E - TS)}{\partial\alpha_0}\right)\left(\frac{\partial\alpha_0}{\partial\alpha_1}\right) + \left(\frac{\partial(E - TS)}{\partial\beta_0}\right)\left(\frac{\partial\beta_0}{\partial\alpha_1}\right) + \left(\frac{\partial(E - TS)}{\partial\beta_1}\right)\left(\frac{\partial\beta_1}{\partial\alpha_1}\right) \quad (65)$$

$$0 = \frac{\partial(E - TS)}{\partial\beta_2} + \left(\frac{\partial(E - TS)}{\partial\beta_0}\right)\left(\frac{\partial\beta_0}{\partial\beta_2}\right) + \left(\frac{\partial(E - TS)}{\partial\beta_1}\right)\left(\frac{\partial\beta_1}{\partial\beta_2}\right) \quad (66)$$

where account has been taken of the dependence of α_0, β_0, and β_1 on the independent variables. The evaluation of (65) and (66) is a problem in calculus. Terms such as $\partial\beta_1/\partial\alpha_1$ or $\partial\alpha_0/\partial\alpha_1$ are computed from Equations (60)–(62); terms such as $\partial(E - TS)/\partial\beta_1$, from (26) and (29). When the indicated calculation is completed, Equation (65) reads

$$\mu^{(G)} = (1 - Z)[(\epsilon_B - \epsilon_A) + kT(\ln\alpha_1 - \ln\alpha_0)] + Z[(\epsilon_{AB} - \epsilon_{AA}) + kT(\ln\beta_1 - \ln\beta_0)] \quad (67)$$

and (66) becomes

$$0 = (Z/2)[\epsilon_{AA} + kT\ln\beta_0 - 2(\epsilon_{AB} + kT\ln\beta_1) + \epsilon_{BB} + kT\ln\beta_2] \quad (68)$$

By taking antilogarithms on both sides of Equation (68), one recovers Equation (12b) as well as the interrelation (14). Equation (67) may similarly be shown to be equivalent to (13) after recognizing that $\mu^{(G)} = \mu_0^{(G)} + kT \ln p$. Upon making this substitution it emerges that the equilibrium constant K is given by

$$kT\ln K = -\mu_0^{(G)} + (1 - Z)(\epsilon_B - \epsilon_A) + Z(\epsilon_{AB} - \epsilon_{AA}) \quad (69)$$

Appendix B; Simplification of the First Order Case

We wish to obtain an expression for F, Equation (37), under the assumption that $w/kT = (\epsilon_{BB} + \epsilon_{AA} - 2\epsilon_{AB})/kT \ll 1$, i.e., that $C - 1 \to 0$. For this purpose, rewrite Equation (12b) as

$$(\alpha_0 - \beta_1)(\alpha_1 - \beta_1)/\beta_1^2 = C \quad (70)$$

which is a quadratic equation in β_1 with the solution

$$\beta_1 = (B - 1)/2(C - 1), \quad B = \sqrt{1 + 4\alpha_1\alpha_0(C - 1)} \quad (71)$$

In view of the assumption concerning w/kT, we can expand the exponential term as $e^{-x} = 1 - x + x^2/2 - \ldots$. Hence

$$C - 1 = e^{-w/kT} - 1 \approx 1 - \frac{w}{kT} + \frac{1}{2}\left(\frac{w}{kT}\right)^2 - 1 = -\frac{w}{kT}\left[1 - \frac{1}{2}\left(\frac{w}{kT}\right)\right] \quad (72)$$

Likewise, the square root can be expanded as $\sqrt{1 + x} = 1 + x/2 - x^2/8 + \ldots$, whence

$$B = 1 + 2\alpha_0\alpha_1(C - 1) - 2\alpha_0^2\alpha_1^2(C - 1)^2 \quad (73)$$

Thus,

$$\beta_1 \approx \alpha_0\alpha_1 - \alpha_0^2\alpha_1^2(C - 1) \quad (74)$$

and

$$\alpha_0 - \beta_1 = \alpha_0[1 - \alpha_1 + \alpha_0\alpha_1^2(C - 1)] = \alpha_0^2[1 + \alpha_1^2(C - 1)] \quad (75)$$

Hence, keeping in mind that $\ln(1 + x) = x + \ldots$, we have

$$\ln(\alpha_0 - \beta_1) = 2\ln\alpha_0 + \ln[1 + \alpha_1^2(C - 1)] \cong 2\ln\alpha_0 + \alpha_1^2(C - 1) \quad (76)$$

Similarly,

$$\ln(\alpha_1 - \beta_1) = 2\ln\alpha_1 + \alpha_0^2(C - 1) \quad (77)$$

When these last two relations are substituted in Equation (37) one obtains

$$F = L\{(Z/2)[\alpha_0\epsilon_{AA} + \alpha_1\epsilon_{BB}] + (1 - Z)(\alpha_0\epsilon_A + \alpha_1\epsilon_B)\} + kTL\{(Z/2)[\alpha_0\alpha_1^2(C - 1) + \alpha_1\alpha_0^2(C - 1)] + \alpha_0\ln\alpha_0 + \alpha_1\ln\alpha_1\} \quad (78)$$

We can simplify as follows:

$$\alpha_0\alpha_1^2(C - 1) + \alpha_1\alpha_0^2(C - 1) = (C - 1)\alpha_0\alpha_1 \approx -\alpha_0\alpha_1 w/kT \quad (79)$$

When this result is used in Equation (78), one obtains Equation (39).

The following result was also required in the section on first order approximation applied to binary mixtures. In simplest terms, Equation (74) reads

$$\beta_1 = \alpha_0\alpha_1 \quad (80)$$

Hence

$$\beta_0 = \alpha_0 - \beta_1 = \alpha_0 - \alpha_0\alpha_1 = \alpha_0^2 \quad (81)$$

$$\beta_1 = \alpha_1 - \beta_1 = \alpha_1 - \alpha_0\alpha_1 = \alpha_1^2 \quad (82)$$

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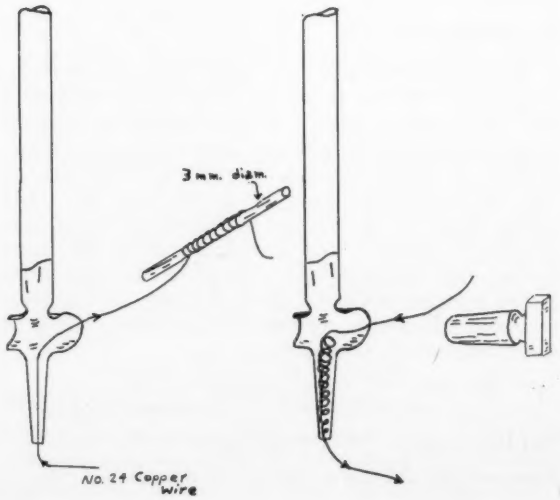
University of South Dakota
Vermillion

A Simple Method for Cleaning Buret Tips

The problem of cleaning buret tips has plagued students, teachers, and chemists for quite some time. The following procedure has been found to be simple, rapid, and efficient for the cleaning of greases and dirt from the tip.

A length of No. 24 copper wire (18–20 in.) is inserted through the tip and out the stopcock housing as shown in the diagram. The wire is then wound around a piece of 3 mm glass rod. After coiling, the rod is removed and the wire pulled through from the tip end (see diagram). As the wire is pulled through the tip, the copper coils partially unravel while the twisting and turning action scrape *all* sides of the tip as the wire is withdrawn.

Upon completion the wire can be wiped clean, and straightened out on the edge of a bench or table for reuse. The procedure is advantageous for silicone greases which do not dissolve in solvents.



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Ada

A Redefinition of "Mole"

A term is a concise, symbolic representation of a concept. Just as individual concepts may be organized to form an idea, so terms may be combined to convey it. Sound terminology promotes *effectiveness* and *convenience* in speech and writing and, through them, comprehension of knowledge. Unsound terminology, on the other hand, impedes communication. Therefore, it is desirable to investigate chemical terminology with the aim of seeking greater comprehension by students. This paper will deal with the investigation and the proposed improvement of a set of terms having the most fundamental significance in chemistry—the gram formula weights.

The requirements of sound terminology may be set forth as follows:

A term should have a *name* which is simple and distinctive;
A term should represent a *concept* which has basic significance (so that it may readily be combined with other terms to convey more complex concepts);
A term should have a *definition* which covers fully the concept it represents.

Although the adjectives "simple," "distinctive," and "basic" in the above requirements cannot be exactly defined, their meanings are quite clear in practice. The human intellect which demands that knowledge be rational also requires that terminology be sound. Either consciously or subconsciously, man is constantly reevaluating the terms with which he is in contact—in common usage, teaching, and learning. Those terms that are unsound will be found *unduly difficult to understand* or *inconvenient to use*. In the inevitable evolution of language, they will sooner or later be revised or simply go out of use.

The terms *gram atomic weight, gram molecular weight, gram ionic weight,* and *gram equivalent weight*—generally referred to as gram formula weights—are most essential to chemical calculations. They are practical, measurable quantities which bear the same proportion to the weight of their respective unit particles. Their long names have given way to many abbreviated forms, such as *mole, g mole, gmw, g atom, g ion, g equivalent, g eq wt, gew,* and simply *equivalent*. But there is lack of uniformity among authors. Their definitions are straightforward, but beginners nevertheless find them hard to grasp. The most difficult appears to be gram equivalent weight.[2] But even in the case of the mole, the student's ability to use the term correctly may not mean full comprehension. An interesting fact is that the mole has frequently been applied to ions in addition to molecules, which shows a tendency to simplify the terminology. The widespread difficulty with, and the evolution in, the gram formula weights are the result of bad terminology as can be shown below.

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Editor's note

Readers will recall the definitive discussion of the "mole" by Professor Guggenheim in THIS JOURNAL, **38,** 86 (1961). *The articles and letters here presented (pp. 549–56) are representative of the communications submitted to the Editor's office since February. This collection is not published with the intention of deciding any pedagogic issue or of establishing unequivocal terminology. The purpose is to urge teachers to take a fresh look at what they have been taking for granted. When skeptical reexamination replaces the habit of mere repetition, teaching improves.*

Reexamining Existing Terminology

A gram formula weight is generally defined in textbooks as "the sum of the atomic weights of a formula, expressed in *grams*," or somewhat more completely, "the quantity of a substance having a weight in *grams* equal to the sum of its atomic weights" (italics mine). Objections may be raised as to (1) the inadequacy of the definition and (2) the lack of "basic" significance of the terms:

(1) As defined, a gram formula weight is merely a weight or quantity, e.g., 1 mole of NO is 30.008 grams, 3 moles of it are 90.024 grams. Actually a gram formula weight is more than that: it is a *unit* of weight or quantity, as in *3 moles of NO*, and *1.70 g atoms of C*, where one mole and one g atom are the respective units. A definition which does not show this unit significance is inadequate; it would be analogous to the definition of meter simply as 39.37 inches long without showing its significance as a *unit* of length.

(2) Again, as defined, 1 g atom of oxygen is 16 grams, 1 mole of water is 18.016 grams, and there are as many different gram formula weights as there are substances. Since each gram formula weight is also a unit, there are, then, as many different units as there are substances. The result is a multiplicity of units which does not appeal to the scientific mind.

This multiplicity of units may be prevented if the definition of a gram formula weight is not based on weight, but on a common denominator among these units. Specifically, there is a number, Avogadro's number, which underlies *all* gram formula weights. Avogadro's number may well be used directly, rather than simply implied, as the basis for the definitions of the gram formula weights, so that all of them will have the same unit.

Some authors, among them Keenan and Wood,[3]

[2] MELDRUM, WILLIAM B., J. CHEM. EDUC., **32,** 48–51 (1955).
[3] KEENAN, C. W., AND WOOD, J. H., "General College Chemistry," 2nd ed., Harper & Brothers, Publishers, New York, 1961, p. 111.

have defined the mole as "*the weight* in grams of 6.02×10^{23} molecules of any substance" (italics mine). Although the numerical significance is explicit, this mole is still a weight, which varies from substance to substance and does not solve the problem of the multiplicity of units. This problem, however, can be solved by defining the mole directly as a pure number.

The apparently simple way in which the terms for the gram formula weights were coined has given rise to names which appear illogical and tend to mislead. Each of the gram formula weights consists of three words—"gram," the word for the species ("atomic," "molecular," ...), and "weight." These three words, taken individually or as a group, may be criticized.

First, the words in gram formula weight are completely separate and hyphens are seldom used; yet they are really dependent parts of a single term. A metric or English unit usually consists of one word (kilogram, gallon) or two words (square meter), but seldom more than two. A unit as simple and frequently used as the one in question should not be represented by three words.

Second, since the word "gram" already implies weight, the presence of "weight" is redundant. Furthermore, no other weight unit carries the word "weight" in the unit ("hundredweight," which is one word, is not an exception). Most serious of all, one might logically ask: If "gram" and "weight" are present in a gram formula weight, why is it not always *explicitly* expressed in grams? The fact that the term is most frequently used as a *unit* is thus made obscure by its very name.

Third, the presence of *atom*, *molecule*, or *ion* in a gram formula weight may also be objected to. They are as unnecessary as the presence of *solid*, *liquid*, and *gas* would be in gram or milliliter. Only when the type is omitted can a term have sufficient breadth to cover all types.

There is a prevalent malpractice in the use of gram formula weight without clearly specifying whether (*a*) the weight in grams or (*b*) the number of the unit is meant. Following is an example of this ambiguous usage taken from a current, widely used textbook:

(*a*) *gram equivalent weight* = gram molecular weight/change in oxidation number,
(*b*) *gram equivalent weights* = normality × liters of solution.

The lack of clear specification is a frequent source of confusion for the beginner.

Some authors have adopted the practice of omitting *weight* for cases like (*b*), while retaining it for cases like (*a*). In similar manner, *g atom* and *g ion* are distinguished from *g atomic weight* and *g ionic weight*. This practice of distinguishing between the complete and the abbreviated names is illogical and tends to confuse the terminology.

Redefining "Mole"

Three conclusions may be drawn from the above:

The set of gram formula weights should not contain the three parts ("gram," the species, and "weight") and therefore should be abolished.

The gram formula weights should be replaced by a single term which is applicable to all species.

The new term should be defined on the basis of Avogadro's number (rather than on weight) and its significance as a unit should be clearly stated.

For the name of this new term, a word derived from Count Amadeo Avogadro's name, such as *Avnola* (Avogadro's no. of formulas) might be adopted; this would also give recognition to a great pioneer in chemistry. However, the use of the term *mole* (which has long been used to imply Avogadro's number of molecules and frequently charged particles) is preferable, for its name has none of the shortcomings of the complete name, and its adoption would prevent dislocations in usage.

The writer proposes to redefine the mole as "*a unit of number equal to Avogadro's number.*" It may be applied to all chemical species (atoms, molecules, ions, radicals, electrons) as well as all physical species (unit cells, colloidal particles, etc.). Examples of correct uses are as follows: 2 moles of copper (or Cu), water (or H_2O), and iodide (or I^-); 2 moles of copper atoms, water molecules, and iodide ions (meaning $2 \times 6.024 \times 10^{23}$ of each kind). If the name of a substance can mean more than one form, as *oxygen* (O_2, O, O^{16}, O^{17}, etc.), the form should be specified; for example, 2 moles of oxygen molecules, atoms, O^{16}, O^{17}, etc. In the absence of an indication, however, the most common molecular form (or isotopic mixture) or the simplest formula should be assumed: 1 mole of chlorine will mean 1 mole of the natural occurring isotopic mixture of Cl_2. Note that it is preferable to say 1 mole of oxygen (6.024×10^{23}) atoms *weighs* rather than *is* 16 grams. The mole as redefined is similar to the words *hundred* and *million* which are units of pure numbers.

Recently, Guggenheim[4] defined the mole as "*the amount of substance* containing the same number of molecules (or atoms or radicals or ions or electrons as the case may be) as there are atoms in 12 grams of ^{12}C" (italics mine). (The actual value of Avogadro's number is not material to the discussion.) This definition recognizes the mole's use in connection with all chemical species, but it cannot be used in connection with a physical species. Another difference between Guggenheim's and the writer's moles lies in the fact that the former is *an amount of substance* which carries with it physical significance, while the latter is entirely *an abstract number*. The question arises, therefore, as to whether it is desirable to attach physical significance to the mole. Since the type of substance should always follow the mole (or should be implied), it does not seem necessary for the mole itself to carry any physical significance.

"Molic" Weight

The weight of a g atom, g mole, or g ion may be expressed as "the weight per mole" of the respective species; e.g., the weight of calcium is 40.06 grams per mole. The frequent use of the concept of *per mole*, however, requires a shorter term which the writer wishes to introduce as "molic." (*Molic* is distinguished from *molar* and *molal* which represent concentration.) Examples of its use are: the molic weight of aluminum is 26.98 grams; the molic volume of hydrogen gas (or any gas) is 22.4 liters under standard conditions. Note that "molic," like "mole," refers to the most common molecular form (or isotopic mixture) or the simplest

[4] GUGGENHEIM, E. A., J. CHEM. EDUC., **38**, 86–87 (1961).

formula if the exact species is not shown. Other examples are: the molic *atomic* weight of nitrogen is 14.008 grams; the molic *ionic* weight of chloride is 35.457 grams; and the molic *molecular* weight of a substance equals the sum of its molic *atomic* weights. The new mole and molic weight are designed to replace gram atomic, gram molecular, and gram ionic weights in all their numerical implications and weight uses.

"Eq" and "Equic"

The related term *gram equivalent weight* should be renamed simply "eq" (to be pronounced as *ek*) which, similar to "mole," will be defined numerically. An eq of a species may be defined as "a unit of number of that species capable of donating, accepting, or sharing one mole of electrons, protons, or electron pairs in a reaction (depending on whether a reductant, oxidant, acid, or base, and whether the Lewis sense is referred to)."

An eq should *always* be related to a mole so that it is convenient to use. For example: 1 mole of zinc is capable of donating 2 moles of electrons and, therefore, is equal to 2 eqs. It follows that 1 eq of zinc weighs one-half of the molic weight ($^1/_2 \times 65.38 = 32.69$ grams). The weight of a substance per eq may be termed "equic weight."

Since the capacity of a substance in donation, acceptance, or sharing may depend on the nature of the reaction, it may be necessary to specify the reaction so that an eq will be a definite number. In this connection, the terms "acid eq," "base eq," "oxidant eq," and "reductant eq" may be used. Thus, 1 mole of H_2CrO_4 is equal to 2 acid eqs or 3 oxidant eqs. The term "milli-eq" may be used to represent a number which is one one-thousandth of an eq.

A solution has been worked out in this paper in simplifying and rationalizing the set of gram formula weights: the writer proposes that the new terms "mole," "eq," and their companion terms "molic" and "equic" weights be adopted to replace the existing set of gram formula weights. *Mole* and *eq* represent very large numbers which cannot be directly measured like the gram formula weights, but the replacement of a weight by a number—of the concrete by the conceptual—follows a well-traveled path in the evolution of terminology.

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The Mole in Quantitative Chemistry

The purpose is to show how the mole concept can be applied more consistently and efficiently to all aspects of quantitative chemistry than conventional approaches that dispense with this concept. Partly to limit the field to be covered, but mainly to show to what extent such well-known laws as those of Dulong and Petit and of Faraday are independent of the molecular hypothesis of Avogadro, this will be done with almost no reference to the relationship of the mole to the molecule, which is discussed elsewhere (*1*). The mole is here regarded as a unit quantity (*2*), in terms of which quantitative chemistry receives its simplest expression, and the conventions of quantity calculus (*3*) are accordingly employed. Some long-established terms will be shown to be either misnomers [like "atomic weight" (*2*)] or superfluous [like "normality" (*4*)]. It is proposed that such terms should be abolished from the teaching of chemistry and replaced by the molar treatment to be described, but supplemented by molecular interpretations outside the scope of this essay.

Origin of the Mole Concept

The mole concept originated in the study of gases (*5*). The molar [or mol (*1*)] amount, n, of any gas, which may be a mixture, is given by:

$$n = \frac{1}{RT} \lim_{P \to 0} PV \qquad (1)$$

The unit in which n is measured is the mole. Suppose that (1) is used to measure the molar amounts, n_i (i = 1, 2, ...), of gases, which are then mixed, and that the molar amount of their mixture is then determined, again using (1). Then it is found that:

$$n = \sum n_i \qquad (2)$$

which may be called the law of conservation of molar amounts (*5*). For ideal gases, (*2*) leads to the law of partial pressures (*6*) and to the law of partial volumes (*7*).

Molar Masses of Solvents and Solutes

The molar [(or proper (*2*)] mass of a substance is the mass of one mole. The molar masses of solutes which appear in the quantitative expressions of the laws of Van't Hoff for extremely dilute solutions and the molar masses of the solvents and solutes which appear in the quantitative expression of Raoult's law are molar masses in the gaseous state (*8*). In the absence of further knowledge, which is molecular and so excluded from consideration here (*1*), it follows that use of either Van't Hoff's laws or Raoult's law will give values for molar masses of solvents and solutes in the gaseous state (*9*).

Based upon a talk entitled "Gulliver in the Land Without Atomic Weights," given on March 25, 1961, at a Short Course for Teachers of Chemistry, at Bradford Institute of Technology, England.

In a recent article in THIS JOURNAL, the statement was made that:

> . . . the fundamental requirement for the determination of atomic or molecular weights is that one be able to count the atoms or molecules, or at least be able to measure equal numbers of atoms or molecules for comparison. This is the principle involved in the measurement of the gram molecular volume for gases and of the freezing-point depression for soluble non-ionizing liquids and solids. The Law of Dulong and Petit must also involve a counting of atoms (*10*).

The term "fundamental requirement" is open to question. That no such fundamental requirement exists is proved by the outline given above of some means of measuring molar masses, all of which are independent of counting molecules, of pairing off sets of molecules, or even of the discovery of the existence of molecules. The importance of the molecular approach is not underestimated, but it seems to be necessary to point out that in cases of the kind cited it is not a fundamental requirement. The reference made to the law of Dulong and Petit (*10*) will be considered later.

Minimolar Masses of Elements

Define the minimolar (or least proper) mass of an element M_{El} as the least mass of an element to be found in its own molar mass, M_{Element}, and in the molar (or proper) masses of its compounds. This definition expresses, in new terminology, the great principle of Cannizzaro for the determination of "atomic weights" (*11*). Minimolar masses are needed to establish the result that the minimolar heat capacity of a solid element, at ordinary temperature and pressure, is about 6.2 cal/deg mole, which is a statement, in molar terminology, of the law of Dulong and Petit (*12*). This is certainly not a law which necessarily involves a counting of atoms, as has been claimed (*10*). From the minimolar masses of the elements which form a compound and the molar mass of the compound it is possible to obtain the usual chemical formula and to realize its molar significance.

Faraday's Laws and the Mole Concept

Faraday's laws (*13*) are expressible in terms of the mole concept by means of the equation (*14*):

$$Q = znF, \qquad (3)$$

in which n is the molar amount of an electrolyte

$$\left(R^{z/\omega_+}_{\nu_+/\omega_+}\right)_{\omega_+}\left(X^{-z/\omega_-}_{\nu_-/\omega_-}\right)_{\omega_-}$$

where z, ω_+, ω_-, ν_+, ν_-, ν_+/ω_+, ν_-/ω_-, z/ω_+ and z/ω_- are positive integers, of which ω_+ and ω_- are coprime, Q is the quantity of electricity involved in the electrochemical reaction of the molar amount n of electrolyte, and F (the Faraday constant) is the least quantity of electricity ever observed to be concerned in the electrochemical reaction of one mole of any electrolyte.

$$\left(R^{z/\omega_+}_{\nu_+/\omega_+}\right)_{\omega_+}\left(X^{-z/\omega_-}_{\nu_-/\omega_-}\right)_{\omega_-}$$

This characterization of F, as what may be called a minimole of positive electricity, is another application of the principle of Cannizzaro (*11*). It is not necessary, in order to formulate the laws of electrolysis, to use "gram equivalent weight" as a variable; equation (3), in which the mole concept is used, can replace F. C. Strong's equation (3), in which the "equivalent concept" is used (*15*).

Chemical Formulas and the Mole Concept

The molar masses of salts cannot be measured by any of the methods which have been described in this account and so it is not possible, in the absence of other information, to ascribe chemical formulas to them. In such dilemmas, empirical formulas are introduced. An empirical formula expresses the composition of a compound in terms of integral minimolar masses of its elements, such that the greatest common divisor of these integers is unity. Sometimes, in the case of a salt, electrochemical evidence demands a multiple empirical formula, like Hg_2Cl_2. The formulas

$$R^{z/\omega_+}_{\nu_+/\omega_+} \text{ and } X^{-z/\omega_-}_{\nu_-/\omega_-}$$

can be called charged chemical formulas, their charge numbers or electrovalences z/ω_+ and $-z/\omega_-$ indicating that their molar (or proper) charges are Fz/ω_+ and $-Fz/\omega_-$, respectively. The evidence that charged chemical formulas are not empirical may be either molar or molecular, but is not considered further here.

Just as a chemical formula is used to designate one mole, an empirical or multiple empirical formula can also be used in this way. As I have shown in more detail elsewhere (*4*, *14*), the citation of any kind of formula can always make evident what a mole is intended to signify in any given context. Thus, a potassium permanganate solution of molar concentration 0.1 M $KMnO_4$ is one whose "normal concentration" is $0.1z$ N-$(KMnO_4/z)$, where z is a positive integer. Again, a calcium fluoride solution whose molar conductance is 196.0 cm^2/ohm mole CaF_2 is one whose "equivalent conductance" is 98.0 cm^2/ohm mole $(CaF_2/2)$. It is apparent that terms like "equivalent weight," "equivalent," "equivalent formula," "equivalent conductance," "normal concentration," and the symbol N for "equivalent/liter" are unnecessary and are better replaced by molar mass, mole, chemical formula, molar conductance, molar concentration and the symbol M for mole/liter, respectively.

There follows, as an example of molar stoichiometry, a problem (*16*) stated and solved in its terms. Although a large number of candidates attempted its solution, the examiner has reported that scarcely a single satisfactory answer was received. The important thing is that the examiner attributes this to a slavish application of "normalities" and "equivalents." Here, then, is ample evidence that understanding based upon the customary emphasis, in the British Isles at any rate, on the "equivalent concept," can be grossly inadequate for dealing with straightforward examples in quantitative analysis.

Twenty-five millilters of a solution containing 23.41 g $MnSO_4$, $4H_2O$/liter were found to react with 29.80 ml of a solution of $KMnO_4$ (A) in the cold and in the presence of excess of sulfuric acid, to yield a red solution. The color of this solution was completely discharged by 34.50 ml of a solution (B) containing 37.24 g $FeSO_4$, $(NH_4)_2SO_4$, $6H_2O$/liter. In a separate titration

25.00 ml of (B) were found to be equivalent to 21.60 ml of solution (A). Use this information to determine the oxidation state of manganese in the red solution, and interpret the reactions involved in the above experiments. (M_{El} mole g^{-1}/El: 1.008/H; 14.01/N; 16.00/O; 32.07/S; 39.10/K; 54.93/Mn; 55.85/Fe.)

Since $5\,Fe^{2+} + MnO_4^- + 8H^- = 5Fe^{3+} + Mn^{2+} + 4H_2O$, it follows that:

$$\frac{25.00 \text{ ml } 23.41 \text{ g } Mn^{2-}\text{/liter}}{223.06 \text{ g/mole}}$$

react, in acid solution, with

$$\frac{29.80 \text{ ml}}{21.60 \text{ ml}} \times \frac{25.00 \text{ ml } 37.24 \text{ g } MnO_4^-\text{/liter}}{5 \times 392.17 \text{ g/mole}}$$

That is, 2.623 mmole Mn^{2+} react with 0.6548 mmole MnO_4^-, or 4 mole Mn^{2+} react with 1 mole MnO_4^-. The reactions involved are

$$4Mn^{+2} + MnO_4^- + 8H^+ = 5\,Mn^{+3} + 4H_2O$$
$$Mn^{+3} + Fe^{+2} = Mn^{+2} + Fe^{+3}$$

The Parsimony of Molar Terminology

The extended use of "mole" and "molar," together with the introduction of a few new terms like "proper" (which is optional), "minimole," and "minimolar," on the lines proposed in this essay, could enable the student easily and efficiently to master and coordinate ideas from studies of many apparently different kinds in the various branches of chemistry. Numerous texts and courses in chemistry are littered with many unnecessary ideas and needless terminology. In the table a comparison is made of this needless terminology and of proposed new terminology which could replace it. The table is a good illustration of the principle of parsimony, also called William of Ockham's razor (*17*), according to which entities, or supposed existences, must not be multiplied beyond what is strictly necessary. It can be justly claimed that the mole concept admirably satisfies this principle.

Comparison of Non-Molar and Molar Terminology

Molar terminology	Non-molar terminology
Law of conservation of molar amounts	Dalton's law of partial pressures; Leduc's law of partial volumes
Law of integral combining molar amounts	Laws of conservation of mass, of constant proportions, of equivalent proportions, of multiple proportions, of reciprocal proportions, and of Gay-Lussac
Minimolar mass	Atomic weight, gram atom, gram atomic weight
Mole	Chemical equivalent, equivalent, mole
Molar concentration	Formal, molar, and normal concentrations
Molar conductance	Equivalent conductivity, molecular conductivity, molar conductivity
Molar cryoscopic constant[a]	Molecular lowering of freezing-point
Molar ebullioscopic constant[a]	Molecular elevation of boiling-point
Molar heat capacity	Atomic heat, atomic heat capacity, molecular heat, molar heat capacity
Molar mass	Chemical equivalent, equivalent weight, formula weight, gram equivalent, gram equivalent weight, gram formula, gram formula weight, gram ion, gram mole, gram molecule, molar mass, molecular weight
Molar volume	Atomic volume, gram molecular volume, molecular volume, molar volume
Molarity	Formality, molarity, normality

[a] $K = M_1{}^G RT\bullet^2/\Delta H_1$, $M_2{}^G = K(w_2/w_1)/(T\bullet - T)$. Both K and $(T\bullet - T)$ are either positive (cryoscopy) or negative (ebullioscopy). The unit of K is deg g/mole.

Conclusion

A logical rather than a historical account of the mole concept as a unifying theme for the understanding of stoichiometry has been presented, deliberately without regard to the molecular hypothesis, in order to reveal its strength as a foundation, as well as its limitations. An appeal has been made for the displacement of a great deal of needless terminology by a few new but necessary terms. It is true that some terms which could be discarded, like "atomic weight" and "electrochemical equivalent," have an almost hallowed place in the development of chemistry. An education of the future chemist which shall encompass something of the glories of its history can still be achieved by footnotes in texts, by the aids of enthusiastic teachers, by articles in journals and in the literature of chemical industry, and by the many books that deal specifically with the history of chemistry. But the student of science will always need, before anything else, to become acquainted as thoroughly and as efficiently as possible with those enduring residues of epochs in science, at all times assessed and presented in terms appropriate to the day in which he studies. That unique chemical contribution to science, the mole concept, is certainly still such a residue.

The author wishes to thank the Governors and the Principal of Bradford Institute of Technology for permission to use matter from a talk given at the Institute, the Council of the Royal Institute of Chemistry for permission to adapt a question set in the Institute's Graduate Membership Examinations, and Professor E. A. Guggenheim for helpful correspondence.

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Chem Ed

LETTERS

To the Editor:

One can speak of "the mass of water" or of "the volume of water," but there is no such term available for the number measure which would indicate reference to molecules or moles. The phrase, "number of water," is not good diction. One can say "the number of grains of sand" because grains are units but not "the number of sand," because sand is one substance. The "number of moles" and the "number of molecules" are too specific; they are analogous to the "number of grams" and the "number of kilograms" and not to the generalized concept.

Similarly, the molecular weight of water is 18 awu/molecule and the molar weight is 18 g/mole, but there is no general term to cover both of these or other related ratios such as g/g-atom or awu/ion.

E. A. Guggenheim has recently pointed up one aspect of this linguistic hiatus by suggesting the word *proper* in order to indicate *one mole*, as in "the 'proper energy' of a substance was so many joules per mole" [J. CHEM. EDUC., **38,** 87 (1961)]. However, this still leaves the general problem of the number concept.

I should like to suggest that there is a word in arithmetic which can be taken for use in this connection. As mass and volume are the (generalized) dimensions which the gram and the liter measure, so cardinal number is the dimension which the mole and the unit measure. Cardinality is the general property analogous to mass and to volume. It therefore seems reasonable to suggest that the *cardinal weight* of a substance is a useful general term, meaning the ratio m/n in any desired units. (Just as density is the ratio m/v in any desired units.) Such expressions as cardinal volume, cardinal energy, and so on, immediately follow.

Those numbers in the periodic table then can be called cardinal weights, with units awu/atom, g/mole, or g/g-atom, depending on the application desired and the predilection of the user.

It is not without timidity that I suggest this. Language is not a logical process. It is only usage that makes words and gives them their meanings, so that we must conform to communicate. I merely offer this as a possibility to any who may have been groping for such a terminology, as I have, in the hope either that we will use it and so make it good language or that some better alternative will be proposed.

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To the Editor:

In a recent article in THIS JOURNAL, **38,** 86 (1961), E. A. Guggenheim defines the mole as follows:

> The mole is the amount of substance containing the same number of molecules (or atoms or radicals or ions or electrons as the case may be) as there are atoms in 12 grams of ^{12}C.

I do not take issue with the scientific content of this definition. However, since "mole" and "Avogadro number" are synonymous terms (attempts to differentiate between them must take the major blame for any confusion engendered in students about the mole), the following definition may have more appeal:

> The mole (Avogadro number) is a pure number equaling the number of atoms in 12 grams of ^{12}C.

The mole (Avogadro number) is thus used analogously to the dozen, the score, the gross, etc. The likelihood that the present value for Avogadro's number may from time to time be altered slightly or equipped with additional significant figures does not affect the usage. We may thus have 1 mole (1 Avogadro number) of molecules, of atoms, of radicals, of ions, of electrons, of clicks on a Geiger counter, of scintillations on a spinthariscope, even of words, of sheep, of stars. In each case we mean $6.02278 \cdot 10^{23}$ of the mentioned items. Some of these items have mass, others do not. In this connection let it be emphasized that it is much more accurate to say, for example, "1 mole of $^{12}C^{16}O_2$ molecules has a mass of (or weighs) 43.98982 g" than "1 mole of $^{12}C^{16}O_2$ molecules *is* 43.98982 g."

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To the Editor:

Your February, 1961, editorial and E. A. Guggenheim's paper on page 86 of the same issue have prompted me to unburden myself of a few ideas on the same subject. The pedantry connected with the term *mole* has always been a bother in classroom presentation and discussion. I believe the difficulty is due to the lack of a generic term to cover the general concept, so well expressed in Professor Guggenheim's definition. I propose that his definition be modified slightly in the manner suggested below and that the term be supplemented by subsidiary terms applying to specific usage.

> The *Avogadro* (avo) is the amount of substance containing the same number of "molecular species" as there are atoms in 12 grams of ^{12}C.

Though some of these "molecular species" exist only in the mind they are real enough in practice, and it might be useful to have terms for them—for example, analytical results such as P_2O_5. For Professor Guggenheim, the term mole would be sufficient, but the rest of the old terms (gram molecular weight, gram atomic weight, gram formula weight, etc.) will die hard and it might be better to tie them to a more general concept rather than have them sulk in confusion among the pages of freshman texts like disinherited relatives.

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Moles and Equivalents: Quantities of Matter

There are three natural methods of measuring out or specifying a quantity of a material: mass, volume, and number. This is readily illustrated by a pound of butter, a quart of milk, and a dozen eggs. The fact that number units are not quite uniform for such things as eggs sometimes limits the effectiveness of number, but in chemistry, where we have such precisely identical units as atoms and molecules, number has become a useful measure of quantity. However, chemists are not in universal agreement concerning the usage and definitions of the number unit, the mole, and a related measure, the chemical equivalent.

The Mole

A *mole*, in the most general usage of the word, is a quantity of something that contains Avogadro's number of units. The units may be atoms, molecules, ions, ion sets, electrons, groups, or whatever else may be of interest, and Avogadro's number is the number of carbon atoms in 12 g of ^{12}C (*1*). The following quotations illustrate this usage.

> To a solution containing n_1 mols of water and n_2 mols of salt . . . (*2*).
>
> The group energy of the methyl group is . . . −338.3 kcal per mole (*3*).
>
> [The bond energy] $D(C—C)$ is equal to 83.6 kcal per mole (*4*).
>
> Here N is the number of moles of electricity passed . . . (*5*).
>
> The complex of one mole of sodium fluoride with two moles of triethylaluminum . . . (*6*).
>
> Pitzer has calculated the following parameters of strain energy associated with bond conformations of three types: staggered = 0 . . ., skew = 0.8 kg-cal/mole; eclipsed = 3.6 kg-cal/mole (*7*).

This usage of the word *mole*, although widely encountered and quite clear, is not the only one found. Some writers prefer to limit use of the word to its original meaning, a collection of *molecules*. When the occasion arises, these writers use such other terms as *gram-atom*, *gram-ion*, and *gram-formula*. Although there is nothing at all incorrect about these other terms, they are not necessary nor must they be used in place of *mole;* they are merely alternatives. Words grow, and chemistry is replete with words which have wandered far from their etymological birthplaces. Consider *atom*, *chromatography*, and *oxidation*. It seems unreasonable to deny the same development of vigor and usefulness to the word *mole*.

On the other hand, use of the special alternatives to *mole* (*gram-atom*, etc.) does have disadvantages: they would seem to make necessary the invention of such dubious terms as *gram-electron*, *gram-bond*, *gram-group*, *gram-group-interaction*, and so on, to fit every special case that arises; they illogically single out one term, the gram-molecule, for the special name of *mole;* and they imply mass rather than number.

There are chemists who consider the mole as a (variable) unit of mass. In this viewpoint, the value of a mole is determined by taking the formula weight and writing *grams* after it.[1] It takes considerable explanation to justify such an apparently high-handed procedure to students, and the procedure is in fact *contrary to experiment*. For example, we do not determine the molecular weight of phosphorous pentoxide or of a protein by adding up a formula-full of atomic weights but rather by dividing an experimentally determined mass by an experimentally determined number of particles. Thus, the molecular weight in practice is defined by the number of moles and not vice versa. One of the earliest demonstrations of this is Williamson's historic work on ethers, in which the formula of water follows from experimental evidence concerning the number of particles:

> Some chemists may perhaps prefer doubling [the formulae] . . .; but I have not felt myself justified in doing so, because that would involve doubling the usual formula for water; for, as I will presently show, water is formed in aetherification by replacing the carburetted hydrogen of alcohol by hydrogen, which, of course, obliges us to assume the same unity of oxygen in both (*8*).

Considering the mole as a unit of mass is in any event contrary to the intended meaning of the word, for the impression we wish to convey and the chemical point at issue in such expressions as *equimolar*, *mole ratio*, and *mole fraction* is one of numbers of units: mass is irrelevant.

For the sake of a thorough-going dimensional treatment of chemical problems, it is helpful to consider the mole as an independent unit, on a par with the gram or the liter. This is not a logical necessity, since the word could have been defined as a pure cardinal number (dimensionless). *Seven*, for example, is such a pure number, with no physical referent. But we use *mole* to refer to a physical collection of objects: when we speak of a mole of oxygen, as well as when we speak of a gram or a liter of oxygen, we refer to a quantity of material and not to an abstract concept or property. Thus, it is our actual use of the word *mole* that makes it dimensionally equivalent to the gram or the liter.

The best usage of *mole* is therefore the most general one: a mole is a quantity of a material containing Avogadro's number of units.

The Equivalent

In addition to mass, volume, and number as the three primary methods of expressing or measuring a quantity of a material, there is a fourth method, the chemical equivalent. This is *a quantity of a material that is related*

[1] It is sometimes said: "The molecular weight expressed in grams." Taken literally, such definitions are absurd, for the molecular weight of water, expressed in grams, is about 3×10^{-23} grams per molecule.

through chemical reaction to one-fourth of a mole of ^{12}C. Alternatively, the definition may be in terms of exactly three grams of ^{12}C. This is no more arbitrary, though less familiar, than the current use of "eight grams of oxygen" or as it rarely appears, "one-half mole of oxygen atoms."

The three-gram standard must refer to the rest mass so that the equivalent, like the mole, is non-relativistic. This is necessary because the chemical reaction, which defines the equivalent, occurs particle by particle and not gram by gram. The mass and volume of a highly accelerated system may suffer relativistic changes but the number of particles contained and therefore the number of moles and number of equivalents do not. Thus, although the observed mass of a mole of accelerated protons may be greater than 1.0073 g, it is still chemically equivalent to three grams of ^{12}C. This also shows that *mole* and *equivalent* convey meanings which are quite independent of mass.

Units, Ratios, and Problems

As there are two independent units of number (the mole and the individual or unit) and two such units of equivalency (the equivalent and the individual or unit), so there are two units of mass, the gram and the atomic mass unit. The gram may be defined as a quantity of a material having one-thousandth the mass of the standard kilogram whereas the amu is a quantity of a material having one-twelfth the mass of a ^{12}C atom. Since the definitions of the two units are completely equivalent and parallel, it is incorrect to state that either unit is more fundamental than the other or that the amu is "only relative."

V. R. Damerell suggested the definition of an *avogram* as "one gram divided by Avogadro's number" (*9*). This is synonymous with the amu even though the wording of the definition seems to imply that the avogram depends on the gram. The reason that the dependence is illusory is as follows: If N is the number of atoms in 12 g ^{12}C, then where x is the mass of one ^{12}C atom, $xN = 12$ g, and $N = 12\text{ g}/x$. Now the avogram is $1\text{ g}/N$ or $1\text{ g}/(12\text{ g}/x) = x/12$, which is one-twelfth the mass of a ^{12}C atom irrespective of the definition of the gram.

I should like to propose yet another synonym (*10*), the *avogadro*, abbreviated *a*. Use of a special name for the amu would tend to obviate the error among beginners of thinking it to be less "real" than the gram. In any event, it would be advantageous to change the definition of the unit, whatever its name, to "one-twelfth of the mass of one ^{12}C atom."

The four methods of expressing quantity, m, v, n, and e, give rise to the six ratios, m/v, m/n, m/e, v/n, v/e, and e/n, and their reciprocals. A common difficulty in connection with these ratios is lack of recognition of the proper units.

Density is ordinarily well defined as m/v with units such as g/ml. Although it is entirely analogous to the other ratios, it is the only one for which we find general agreement in definitions. The other ratios are used in the same ways, but many definitions are offered.

The simplest and most common use of molar weight is in expressions such as $mw = m/n$ or $m = n \times mw$. Such usage shows that molar weight is the ratio m/n with units such as g/mole, lbs/lb-mole, or amu/unit. This describes the intended meaning in good usage and also makes these equations dimensionally valid.

Similarly, molar volume is v/n with units such as liters/mole, equivalent weight is m/e with units g/equiv, and equivalent volume is v/e with units such as liters/equiv.

The remaining ratio, e/n, which may be called the *empirical valence* and which usually is denoted by the letter z, is almost always shorn of its units, equiv/mole. There is nothing wrong with its usual description as the number of replaceable H^+ or OH^- ions or the number of electrons gained or lost (*per unit*), but the more empirical definition takes care of all possible cases and also provides usable units. G. W. Bennett (*11*) has remarked on the empirical basis of this ratio and its relation to the others, and S. W. Benson (*12*) has given the definition and units derived here.

The use of these ratios with their proper units is not merely a powerful aid in problem-solving but also reflects a fundamental viewpoint. Thus, there is a difference between logical (or pure) mathematics, which deals entirely with abstractions, and physical mathematics, which handles physical quantities and their ratios. In the latter case, a quantity is treated as the product of an abstract number and a dimension. Dimensions are multiplied, divided, and so on, according to the rules of pure mathematics. The advantage of using pure mathematics, which also has been called "quantity calculus," has been discussed (*13*). Texts are available which show how this can be well done at the level of beginning students (*12*, *14*, *15*). A few examples here will be enough to show the increased clarity which results from this practice and to show how the definitions given here correctly describe actual usage.

$$m = 10\text{ (ml)} \times 1.56\text{ (g/ml)} = 15.6\text{ g}$$
$$m = 2\text{ (moles)} \times 18\text{ (g/mole)} = 36\text{ g}$$
$$V = 22.4\text{ (l/mole)} \times 2\text{ (moles)} = 44.8\text{ l}$$

$$ew = \frac{98\text{ (g/mole)}}{2\text{ (equiv/mole)}} = 49\text{ (g/equiv)}$$

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(13) Guggenheim, E. A., J. Chem. Educ., **35**, 606 (1958).
(14) Pierce, C., and Smith, R. N., "General Chemistry Workbook," 2nd ed., **1958**, W. H. Freeman, San Francisco.
(15) Compton, C., "An Introduction to Chemistry," D. Van Nostrand Co., Princeton, N. J., **1958**.

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Molecular Weights by Cryoscopy

A general chemistry laboratory experiment

Cyclohexanol appears to be an ideal cryoscopic solvent because of its large depression constant (approximately 39), fairly general solvent properties,[2] and a melting point slightly above room temperature. Several methods[2–4] using this solvent have been described in the literature, but they were found to be unsatisfactory for the general chemistry laboratory because of the techniques involved and the hydroscopic nature of cyclohexanol. The experiment here described has advantages over previously reported methods using cyclohexanol in that the entire system is protected from atmospheric moisture throughout the procedure, it requires only simple equipment (standard 1° laboratory thermometers may be used), and the determinations are rapid. Students with no previous experience with this method can determine the molecular weight of an "unknown" (±8%) in one-half hour providing the cryoscopic constant is known. While this was designed as a general laboratory experiment, it has been employed extensively in our organic and qualitative organic laboratories where the limits of error were reduced to ±2% by using thermometers graduated by 0.1°.

The Experiment

Apparatus. Each student is given a sealed vial of redistilled cyclohexanol (approximately 20 g of solvent), two empty 6-dram (25 × 85 mm) lip vials fitted with corks, a 0–100° thermometer graduated in degrees, and one syringe bottle stopper (rubber, sleeve type, 11A to fit 6-dram vials) bored to allow insertion of the thermometer (see the figure).

Solvents and Unknowns. The cyclohexanol (Eastman Kodak #703) is distilled under reduced pressure at the rate of 10 ml per minute from a simple distillation flask. Material freezing at 24° or higher is collected and placed in vials, which are then stoppered and sealed with paraffin wax. (These vials can be stored for at least six months without detectable change in melting point of the cyclohexanol.) The standards and "unknowns" used were Eastman Kodak White Label and were not further purified.

Determining the Cryoscopic Constant of Cyclohexanol

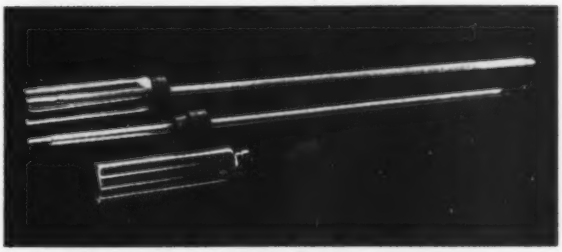
Approximately one-half of the pure cyclohexanol is weighed into one of the empty vials. Enough benzoic acid is then weighed into this vial to make a 2–3% solution by weight. The vial is stoppered and shaken until all of the solid dissolves.

The vial containing the unweighed pure cyclohexanol is fitted with the syringe bottle stopper. The thermometer is inserted through the stopper to within 5 mm of the bottom of the vial (insertion of the thermometer can be facilitated by first lubricating the thermometer stem with a small amount of the solvent). The vial is placed in a beaker of cold (15°) water and its contents stirred with the thermometer until a thick mush is formed, then removed from the beaker and allowed to warm slowly in the hand while stirring is continued. The melting point is taken as the temperature at which the last crystal of cyclohexanol disappears. This procedure is repeated at least three times to determine an average melting point. Usually the observations are within 0.5° of each other.

The syringe bottle stopper and thermometer are then removed from the vial of pure cyclohexanol, wiped clean, and fitted to the vial containing the cyclohexanol-benzoic acid solution. The vial of pure solvent is restoppered and reserved for the second part of the experiment. The melting point of the benzoic acid solution is then determined in the same way as described for the pure cyclohexanol.

The cryoscopic constant, K_f, of the solvent is determined by substituting the weights of solvent cyclohexanol G and solute benzoic acid g, the melting point of the pure solvent ($T°$) and solution (T_1) and the molecular weight of the benzoic acid into the familiar equation

$$M_{\text{solute}} = \frac{1000\ K_f g}{G(T° - T_1)}$$



Molecular Weight of an Unknown

The remainder of the pure cyclohexanol is weighed into the second empty vial. Enough of the unknown is weighed into this vial to make a 2–3% solution by weight. The vial is stoppered and shaken until all of the unknown is dissolved. The syringe bottle stopper

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[1] This paper reports a part of the research project carried out by R. Mikulak during his freshman year at Hamline University.

[2] In general, cyclohexanol is not a good solvent for urea or its derivatives, nitrophenylhydrazones, halogenated compounds, or sugars. See Wilson, H. N., and Heron, A. E., *J. Soc. Chem. Ind.*, **60**, 168 (1941).

[3] Cheronis, Nicholas, "Technique of Organic Chemistry," Vol. 6, Arnold Weissberger, ed., Interscience Publishers, Inc., New York, 1954, p. 213.

[4] Cheronis, Nicholas, and Stein, Herman, "Semimicro Experiments in General Chemistry and Qualitative Analysis," John de Graaf, Inc., New York, 1959, p. 103.

and thermometer used in the first part of the experiment are wiped clean and fitted to the vial, and the melting point of the unknown solution is determined in the same manner as described for pure cyclohexanol. The molecular weight of the unknown is then calculated using the experimentally determined K_f.

The table lists a number of compounds whose molecular weights were determined by freshman chemistry students. Cyclochexanone, cyclopentanone, cyclohexane, cyclohexylamine, benzene, and chloroform were found to be unsatisfactory since they gave consistently high results. The cryoscopic constant of cyclohexanol calculated from latent heat data[2] is 39.6, while that found by this method using benzoic acid, acetanilide, and triphenylcarbinol as standards was 39.3 ± 0.5 (10 trials).

Molecular Weights—Student Results

Compound	Calculated	Found (av)	Number of trials
Acetanilide	135	131 ± 5.1	7
Benzoic acid	122	122 ± 5.0	5
Bromobenzene	157	155 ± 13	4
Benzaldehyde	106	112 ± 3	3
Decanoic acid	186	181 ± 5	2
Anthranilic acid	137	136	1
p-Nitroaniline	138	145 ± 4	3
Anethole	148	151	1
Anisole	108	120	1
Chlorobenzene	102	97	1
Benzophenone	182	173	1
Naphthalene	128	117	1
Water	18	17	1
Triphenylcarbinol	262	260	1

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Analysis of an Aluminum-Zinc Alloy

A general chemistry laboratory experiment

One of the most commonly performed quantitative experiments in general chemistry is the determination of the gram equivalent weight of a metal by measuring the volume of hydrogen evolved in the reaction of a weighed metal sample with excess acid. The experiment as it is ordinarily carried out involves the use of pure samples of aluminum, zinc, and magnesium as "unknowns." Since these metals are rather easily identified by visual observation or simple chemical tests, it was felt desirable to substitute for them a series of alloys of varying composition. Aluminum and zinc were chosen as alloy constituents because of the wide difference in their gram equivalent weights. The analysis of such alloys, in addition to offering a greater challenge to students, illustrates a practical application of the concept of gram equivalent weight and of the use of the gas laws.

The alloys were prepared by melting together pieces of block aluminum and zinc pellets in a clay crucible. The molten mixture was stirred to ensure homogeneity and cooled to just below the melting point, where the two metals form a solid solution. The metal ingot formed was quenched in water, the oxide coating adhering to the surface removed, and the alloy converted to turnings on a lathe. A series of five alloy samples each weighing about 1 kg and varying in composition from 27 to 57% aluminum was prepared.

Sufficient alloy was issued to each student to enable him to weigh out duplicate samples varying in mass from 0.120 to 0.180 g. The procedure followed in the experiment was similar to that described in Selwood.[1] The volume of hydrogen generated was found by determining the weight of water required to completely fill the gas bottle in which the hydrogen was collected.

Each student was instructed to report the per cent of aluminum in his sample, making use of the relations

$$\text{Volume } H_2(\text{STP}) = \text{Wt Zn} \times \frac{11.2\text{ l}}{32.7\text{ g}} + \text{Wt Al} \times \frac{11.2\text{ l}}{8.99\text{ g}}$$

$$\text{Weight sample} = \text{Wt Zn} + \text{Wt Al}$$

A data sheet was submitted with each report so that errors in calculations, of which there were many, could be detected.

The precision of the experiment, determined from results submitted for about 1000 samples, is indicated by data for the alloy containing 57% Al; 34% of the results were within ±1% of the median, 59% within ±3% and 74% within ±5%. The principal errors arise from inaccuracies in weighing the sample and in measuring the volume of hydrogen evolved. It can be calculated that with the apparatus used, an error of 0.001 g in the weight of alloy sample produces an error in the reported % of aluminum of about 0.5%. An error of 0.5 ml in the volume of the hydrogen collected gives rise to a deviation of the same magnitude.

[1] Selwood, P. W., "Experiments in General Chemistry," 3rd ed., Henry Holt and Company, Inc., New York, 1959, p. 25.

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An Easily Prepared Wide Range Buffer Series

Over the years, explaining to students the reactions involving strong and weak acids and bases and their salts, either by lecture demonstration or by laboratory experiment, has been handicapped by the lack of a simple and inexpensive means of indicating the *p*H of the experimental solutions used. The use of a *p*H meter is impractical mainly because of the cost but also because it involves principles of measurement not understood by the student. In recent years the development of mixed indicators capable of indicating the *p*H of solutions over wide ranges has greatly increased the possibility of designing new significant demonstrations and experiments. In his own classes, the writer has found that the introduction of demonstrations and experiments using the mixed indicator of extremely wide range developed by Richardson[1] produces very satisfying results.

One requirement for demonstrations and experiments involving indicators is a wide-range buffer series of *p*H standards for comparison with the experimental mixtures. The preparation of one of the several well-known series of buffer solutions is not a serious problem in the analytical laboratory of a college or university, but under different circumstances it may indeed be enough of a problem to discourage the average high school teacher and many college instructors from undertaking a particular experiment or demonstration. There appears to be a need for a wide-range buffer series that may be prepared with *moderate* accuracy from two or three common chemicals and that requires the use of equipment as available and inexpensive as a graduated cylinder and a laboratory trip scale.

The original buffer series developed by S. P. L. Sorensen[2] requires the use of eight chemicals in seven different stock solutions, including "carefully" standardized solutions of sodium hydroxide and hydrochloric acid. The more commonly used series of Clark and Lubs[3] as well as the wide-range series developed by Prideaux and Ward[4] use fewer chemicals and stock solutions but still require standardized solutions. The well known series of McIlvaine[5] satisfies the requirements in that it uses only two chemicals, two stock solutions and no standardized solution. This series, however, is limited on the alkaline side to buffers with a *p*H of 8.0 or less. Other less known series also fail to meet the requirements in one way or another.

[1] Richardson, F. R., J. Chem. Educ., **33**, 517 (1956).
[2] Sorensen, S. P. L., *Ergeb Physiol.*, **12**, 393 (1912).
[3] Clark, W. M., and Lubs, H. A., *J. Biol. Chem.*, **25**, 479 (1916).
[4] Prideaux, E. B. R., and Ward, A. T., *J. Chem. Soc.*, **125**, 426 (1924).
[5] McIlvaine, T. C., *J. Biol. Chem.*, **49**, 183 (1921).

The goal of the present work was the development of a series of buffers with *p*H values ranging from 2.0 to 12.0 that would require the preparation of only two stock solutions and would use only two or three common chemicals. Also it should be preparable with the desired precision (± 0.05 *p*H units) using ordinary graduated cylinders for measuring the stock solutions. A Model G Beckman *p*H Meter was used in determining the *p*H values of the experimental solutions. A Type E-2 glass electrode was used on the alkaline side. The meter was standardized and checked against buffer standards in the same region of *p*H as that of the particular experimental solution being measured.

The goal has been realized by the use of three common reagent-grade chemicals: boric acid (anhydrous), citric acid (monohydrate), and tertiary sodium phosphate ($12H_2O$), dissolved in two stock solutions. Solution "A" is 0.200 *M* boric acid and 0.050 *M* citric acid. Solution "B" is a 0.100 *M* solution of tertiary sodium phosphate. These stock solutions (one liter or more of each) are prepared by weighing out (to the nearest 0.1 gram) the correct amounts of the components and diluting to volume in a volumetric flask. The volumes of the two stock solutions used in preparing 200 ml of buffer solution at different *p*H values are shown in Table 1.

Investigators over the years have avoided the use of tertiary sodium phosphate as a component of *accurate*

Table 1.—Volumes of Acid Solution "A" and Basic Solution "B" Required for the Preparation of 200 ml of Solutions in Buffer Series

*p*H	Solution "A" volume (ml)	Solution "B" volume (ml)
2.0	195	5
2.5	184	16
3.0	176	24
3.5	166	34
4.0	155	45
4.5	144	56
5.0	134	66
5.5	126	74
6.0	118	82
6.5	109	91
7.0	99	101
7.5	92	108
8.0	85	115
8.5	78	122
9.0	69	131
9.5	60	140
10.0	54	146
10.5	49	151
11.0	44	156
11.5	33	167
12.0	17	183

Solution "A": Boric Acid 0.2 *M*, Citric Acid 0.05 *M*.
Solution "B": Tertiary Sodium Phosphate, 0.1 *M*.

buffer series not only because of the poor quality of the commercially available salt but because of the problem of preparing a product of a definite composition. Bell[6] has obtained some evidence for the existence of two hydrates of the salt mixed with sodium hydroxide. On the other hand, Bates, Pinching, and Smith[7] report that a stable product with a definite water content of between zero and 12 moles per mole of salt would be "difficult or impossible to obtain."

The quality of the commercially available tertiary sodium phosphate has improved materially during the past few years and at the present time at least two manufacturers are furnishing a "reagent grade" salt with the maximum sodium hydroxide content (approximately 2.0%) indicated on the label. To test the consistency of the brands of "reagent grade" tertiary sodium phosphate available, four different sets of buffers were prepared according to the figures given in Table 1 using two lots of one brand manufactured two years apart and one lot each of two brands of other leading manufacturers.[8] The *p*H readings of the four series agreed within 0.03 units throughout.

Buffer Capacity of Mixtures

The seven weak acids involved in this series of buffers are listed in Table 2 according to decreasing ionization constants. As Prideaux and Ward[4] have pointed out, the production of an effective buffer series of wide range depends upon the inclusion of a sufficient number of weak acids differing by relatively small amounts in their *pK*'s so that the effect of successive acids overlap. On the other hand if the *pK*'s of the acids are too far removed from one another the stronger acid is completely neutralized, or nearly so, before the neutralization of the weaker acid commences. It is to be noted that the differences between the successive *pK*'s of the acids in Table 2 are relatively small with the exception of the last two, which differ by 2.8 units. One should expect that a buffer series which involves the acids listed would have consistently good capacity with the possible exception of those buffers with *p*H values in the region approximately half-way between *p*H 9.2 and *p*H 12.0.

[6] Bell, Russell N., *Ind. Eng. Chem.*, **41**, 3901 (1949).

[7] Bates, R. G., Pinching, G. D., and Smith, E. R., *J. Research Natl. Bur. Standards*, **45**, 418 (1950).

[8] Samples were supplied by J. T. Baker Chemical Company, General Chemical Company, and Mallinckrodt Chemical Works.

The buffer capacity of selected buffers of the series was determined by measuring the change of *p*H caused by successive additions of relatively small volumes of standard HCl solution to 100 ml of the buffer solution. The buffer capacity was found to fluctuate between limits of 0.025 and 0.030 moles per liter per *p*H unit for mixtures in the region between *p*H 2.0 and *p*H 10.0. Above *p*H 10.0 the buffer capacity of the mixtures was found to diminish to a low of 0.020 at *p*H 10.5 and then, above *p*H 11.0, to increase rapidly to a high of 0.09 at *p*H 12.0. The addition to the system of an acid with a *p*K in the neighborhood of 10.5 would undoubtedly increase the buffer capacity of the mixtures in this area, but any improvement so gained would not be worth the trouble of buying and weighing out a fourth component for the mixtures.

Table 2.—Ionization Constants of Weak Acids Included in Buffer Series

Acid	K_a	*pK*
H_3PO_4	8×10^{-3}	2.1
$H_3C_6H_5O_7$	8×10^{-4}	3.1
$H_2C_6H_5O_7^-$	2×10^{-5}	4.7
$HC_6H_5O_7^{-2}$	4×10^{-6}	5.4
$H_2PO_4^-$	6×10^{-8}	7.2
H_3BO_3	6×10^{-10}	9.2
HPO_4^{-2}	1×10^{-12}	12.0

Precision Requirements

A consideration of the differences in the volumes of solution "A" and/or solution "B" required to prepare successive buffers, as shown in Table 1, indicates that the *p*H is most sensitive to such volume changes in the range of buffers from *p*H 10 to *p*H 11. At such sensitive points the precision with which the *p*H of a buffer may be reproduced is poorest. In this region a change of 10 ml in the volume of solution "A" corresponds to a change in the *p*H of the buffer solution of 1.0 unit. In order to produce a buffer with *p*H precise to the nearest tenth unit (± 0.05) it would be necessary to measure the volume of the acid to the nearest 1.0 ml. This is possible using a clean 100-ml graduated cylinder. It should be noted that any error in the measurement of solution "B" also introduces an error of the resulting mixture; however, this error would be relatively smaller in this region, since the volume of solution "B" to be measured is from three to four times as great as that of solution "A."

Noted and Quoted

But in four hundred years since the Scientific Revolution we have learned that we gain our ends only with the laws of nature; we control her only by understanding her laws . . . . We must be content that power is the by-product of understanding. So the Greeks said that Orpheus played the lyre with such sympathy that wild beasts were tamed by the hand on the strings. They did not suggest that he got this gift by setting out to be a lion tamer.

J. Bronowski, "Science and Human Values"

Wyndham D. Miles
The National Archives
Washington, D. C.

Robert Maskell Patterson

First to teach atomic theory in America?

When John Dalton of Manchester, England, conceived his atomic theory in the opening years of the 19th century he did not hurry to publicize his ideas. He first mentioned them locally to his scientific friends, to audiences at the Manchester Literary and Philosophical Society, and perhaps to audiences at his lectures elsewhere. In 1804 he discussed his ideas with Thomas Thomson, and Thomson gave them to the world in the third edition of his popular textbook, "System of Chemistry," published in London in 1807. The following year Dalton issued his "New System of Chemical Philosophy" and from then on it was only a matter of time until Dalton's book came into the hands of chemists in Great Britain, Germany, France, the United States, and other countries.

It is hardly possible to discover who first taught Dalton's atomic theory in an European university. Owing to Thomson's missionary work the theory got around quickly in Great Britain, although there was by no means unanimous approval of the theory among British chemists. British professors soon introduced it into their lectures and perhaps it appeared almost simultaneously in a number of educational institutions. Within a few years it was widely spread, as evidenced by references to it in scientific books and articles of the times.

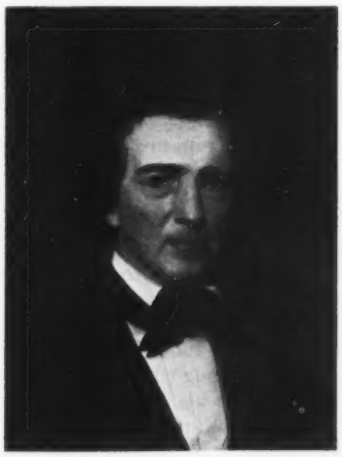
How did the theory reach the United States? Probably first through imported books, including Thomson's "System of Chemistry" and Dalton's "New System." Secondly and thirdly, via the pages of European scientific journals and through Americans who attended chemical lectures in Europe.

Who first taught the theory in the United States? The honor may belong to an American student who was abroad around this time, Robert Maskell Patterson of Philadelphia, son of Robert Patterson, Thomas Jefferson's Director of the Mint and Professor of Mathematics and Natural Philosophy at the University of Pennsylvania.

Young Patterson, born in Philadelphia March 23, 1787, received the Bachelor of Arts and Doctor of Medicine degrees from the University of Pennsylvania, where he had been attracted to chemistry by James Woodhouse, America's foremost chemist of that day; in 1809 he sailed to Europe for further study. He spent the first two years of his tour in Paris. There he attended the lectures of Louis Nicolas Vauquelin, which, in his words, were "known to constitute the most learned and complete course of chemistry given in Europe"; those of Louis Jacques Thenard, whom he called the "most popular lecturer on chemistry in Paris, and the happiest demonstrator, except Dr. Wistar [a professor of chemistry in the Medical School of the University of Pennsylvania] that I have ever seen"; and lectures at the "Garden of Plants" and at the School of Medicine.[1] Along with chemistry he studied mathematics, physics, and medicine.

While Patterson was in Paris the American minister, John Armstrong, asked him to act temporarily as the American consul. When Patterson's name was presented to Napoleon the Emperor exploded, refusing permission for Patterson to take the post because he assumed that the young man was related to Elizabeth Patterson of Baltimore, the girl who had married Napoleon's brother Jerome and whose marriage Napoleon had annulled because of her plebeian ancestry. The storm blew over, however, and Patterson filled the office through the year 1809.[2]

In 1811 Patterson began to retrace his steps homeward. He stopped in London to attend the lectures of Humphry Davy.[3] In Britain he also purchased apparatus and chemicals to take back with him to the States. Finally in 1812 he sailed west across the Atlantic.



Robert Maskell Patterson (from a painting by Samuel R. DuBois, courtesy of the American Philosophical Society).

Back in Philadelphia, Patterson intended at first to practice medicine, but his plans took a turn owing to the poor health of his father. Patterson took his

[1] Testimonials of Robert M. Patterson to the trustees of the University of Pennsylvania, August 4, 1818. In the Archives, University of Pennsylvania.

[2] Breck, Samuel, "Short Biography of Robert M. Patterson, M.D." (Philadelphia, 1854), p. 10. This biography is a 16-page pamphlet.

[3] See footnote 1.

father's place temporarily in the classroom at the University of Pennsylvania and delivered a course of lectures on chemistry. Shortly thereafter the trustees of the University elected him to the professorship of natural philosophy in the Medical School. The course in natural philosophy was not required of the medical students, but was elective. Consequently the vast majority of the embryonic physicians refused to spend their time or money taking the course, and Patterson, whose only remuneration was student fees, could not make a living from this position alone. He might have had to forego teaching and turn to the practice of medicine if other positions had not opened up for him at the University. In February, 1813, the trustees elected him Professor of Natural History,[4] and in September, 1813, they engaged him temporarily to replace his father, who had resigned.[5] The following year they elected him to his father's old professorship.[6]

In stepping into his father's shoes Patterson found himself burdened with a multitude of subjects. His primary responsibility was mathematics, but in addition he was required to teach physics, chemistry, and astronomy. The trustees gave him permission to obtain an assistant in mathematics—provided that Patterson pay the assistant's salary.[7] We have only to glance at the college schedule for 1817 to see the heavy load that Patterson carried. He taught arithmetic, geometry, and algebra to the freshmen; trigonometry, solid geometry, surveying, analytical geometry, algebraic functions, and calculus to the sophomores; calculus, natural philosophy, astronomy, and a "course of lectures on chemistry" to the seniors.[8] In addition he still lectured on natural philosophy to the medical students. Some of the elementary subjects may have required little of the professor's attention outside of the classroom, but the total time taken up by the large number of class meetings did not leave the professor much time for research. Here, in my opinion, is a major reason why Patterson did not go on to make more of a name for himself in American science.

In 1818 came a possible opportunity for Patterson to step into a much more desirable academic post, the professorship of chemistry in the Medical School of the University. The Medical School was the most famous institution of its kind in the country. The number of students ranged into the hundreds, whereas the College, in contrast, seldom carried more than fifty on its roll. The professor of chemistry received an excellent living from student fees, he had space for laboratory work (although he had to provide his own apparatus and chemicals), he concentrated on only one subject and he had time for research.

Patterson solicited testimonials to submit to the Board of Trustees, as did the other candidates for the chair. Professor William G. Horner, one of those who came out for Patterson, wrote that the election was a constant topic among the medical students, the majority of whom were in favor of Patterson because of his great popularity as a lecturer on natural philosophy.[9] Thomas P. Jones, former Professor of Natural Philosophy at the College of William and Mary, said that Patterson was the "best lecturer on the physical sciences that I have ever heard."[10] Joseph Cloud of the United States Mint, a chemist of considerable reputation, said that he had attended two courses on chemistry delivered by Patterson and that the latter was a successful, neat experimenter with an accurate knowledge of the science.[11] Patterson forwarded a résumé of his own qualifications and it was here that he stated that, after he had returned from Europe in 1812, he had delivered chemistry lectures to the seniors at the University and that he believed that those lectures "were the first in which the atomic theory . . . was taught in this country."[12] Patterson put up a good fight, but unfortunately for him he was opposed by two giants, Thomas Cooper and Robert Hare, the latter winning the election and going on to become the greatest American research chemist of his generation.

Now, was Patterson correct in his belief that he was the first to teach Dalton's theory in America? In his favor are these circumstances: he was on the ground; and he was acquainted with the curricula, subject matter, and teachers in other American colleges. It is therefore reasonable to place faith in his statement. On the other hand those of us who are interested in the history of American science would like to have more than his unsupported statement as proof of his assertion. But how are we to prove or disprove Patterson? This is a difficult, perhaps impossible, task. Our primary knowledge of early American chemistry courses comes from notes taken by students. *If* we had continuous runs of notes taken by students who attended John Maclean at Princeton, Samuel Latham Mitchill at Columbia, Benjamin Silliman at Yale, James Woodhouse and John Redman Coxe at the University of Pennsylvania, and other American professors of chemistry between the years of, let us say, 1807 and 1815, we *might* be able to ascertain when Dalton's theory appeared in each college. But alas! Early American student notes are quite rare, and no University has enough of them in its archives to trace the evolution of its early chemistry course. There are other sources of information—college catalogs, minutes of the trustees, letters, biographies, autobiographies, newspaper articles and so on—but they are not nearly as useful or as accurate as student notes in determining the content of an early course. Probably we will never be able to settle this matter definitely.

Where did Patterson learn of Dalton's theory? Humphry Davy appears the most likely source. It could be objected that Davy was not enthusiastic

[4] Minutes, Board of Trustees, University of Pennsylvania, February 3, 1813.
[5] *Ibid.*, September 7, 1813.
[6] *Ibid.*, March 3, 1814.
[7] *Ibid.*, August 6, 1816.
[8] "Rules and Statutes of the University of Pennsylvania" (Philadelphia, 1817). N.p.
[9] Letter, Horner to Patterson, August 1, 1818. In Archives, University of Pennsylvania.
[10] Letter, Jones to the trustees, University of Pennsylvania. In Archives, University of Pennsylvania.
[11] Letter, Cloud to the trustees, University of Pennsylvania, July 29, 1818. In Archives, University of Pennsylvania.
[12] See footnote 1.

over Dalton's ideas, but even so he probably felt it his duty to mention them, along with his own reservations, to the audiences attending his lectures. Other possibilities are Vauquelin, Thenard, and other French chemists whose lectures Patterson attended in Paris between 1809 and 1811. This question, however, is another to which we cannot give a positive answer.

Patterson's chemistry lectures were quite satisfactory, judging by remarks of his contemporaries, but other than that we know practically nothing about the course. The professor never published any textual material for his students. Probably he employed one or more of the standard British or American works in the classroom. We learn, from references in the Minutes of the Board of Trustees of the University to scientific equipment and to expenses for chemistry lectures,[13] that Patterson made use of lecture demonstrations in his course, but the students did not receive any laboratory training. Our ignorance of Patterson's preferred text coupled with a failure to locate notes taken by his students makes it impossible for us to form a conception of the chemistry course that he taught during his tenure at the University.[14]

Patterson remained at the University of Pennsylvania as a professor and Vice Provost until 1828. At that time the trustees reorganized the College, and Patterson, who perhaps was not in complete agreement with the Board, resigned and went to the University of Virginia.[15] Dr. Robley Dunglison of the Virginia faculty once said that "as a lecturer on science, Dr. Patterson was one of the most successful I have ever heard."[16] Patterson remained at Charlottesville for seven years, resigning to accept from President Jackson the directorship of the Mint. From his office in Philadelphia he superintended the operations of the Philadelphia mint and of the three southern branches until he retired in 1851 because of ill health.[17]

Outside of the field of science, Patterson was active in community affairs. In 1813, during the war with Great Britain, the British planned attacks along the coast which led the Americans to erect temporary fortifications around Philadelphia. Patterson supervised some of this work, and received official thanks from the Committee of Safety after the invasion threat had passed.[18] He was one of the managers of the Pennsylvania Institution for the Instruction of the Blind, a President of the Musical Fund Society of Philadelphia, one of the founders of the Franklin Institute (where he also taught natural philosophy), a trustee of the University of Pennsylvania, and president of a life insurance company.[19] It is said that he was asked to run for Congress and declined.[20] He was one of the youngest men elected to the American Philosophical Society, becoming a member when he was twenty-two.

Patterson published little, possibly because he was so busy with professional and civic duties. I have located only one article dealing with scientific matters, and two with history.[21] There are, however, in the *Proceedings of the American Philosophical Society* between 1837 and 1854 a number of "verbal communications" by Patterson, in the nature of abstracts of talks he gave before the Society. One of Patterson's contemporaries said that he did not write much and wronged his memory by not publishing what he wrote.[22]

Patterson died in Philadelphia on September 5, 1854. During his life he was widely known and was respected as an excellent, conscientious teacher and an able administrator. Today he is practically forgotten in American chemistry, but to him may belong the honor of having introduced Dalton's atomic theory into the United States.

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[13] See footnote 1. Also, Minutes, March 2, 1813; August 6, 1816; March 4, 1828; and September 13, 1828.

[14] The only student notes that I have located are those taken by Charles F. Schaeffer in 1827. The notes cover 131 pages, and are devoted to physics. In Historical Society of Pennsylvania.

[15] Minutes, April 1, 1828; May 27, 1828.

[16] *Proceedings American Philosophical Society*, **6**, 9 (1854–58).

[17] Many of the records of the Mint generated during Patterson's administration are in the National Archives, Washington. They are described in Preliminary Inventory No. 40, Records of the United States Mint at Philadelphia, obtainable from the Archives.

[18] BRECK, *op. cit.*, pp. 11–12.

[19] *Ibid.*, p. 12.

[20] *Ibid.*, p. 15.

[21] "Ueber die Beschaffenheit und das Verkommen des Goldes, Platin, und der Diamanten in der Vereinigten Staaten," *Zeitschrift Deutsche Geologische Gesellschaft*, **11**, 60–64 (1850).

"Early History of the American Philosophical Society," *Proceedings American Philosophical Society*, **3**, 3–36 (1843).

"Address of Dr. Robert M. Patterson, at the close of the Thirteenth Exhibition of American Manufacturers, held by the Franklin Institute" *Journal Franklin Institute* [3 series] **7**, 36–46 (1844).

[22] Obituary of Patterson by KANE, J. K., in *Proceedings American Philosophical Society*, **6**, 60–64 (1854–58).

World Wide View of New Ideas in School Chemistry

The Office of Scientific and Technical Personnel of the Organization for European Economic Cooperation (OEEC) held a seminar on "The Status and Development of the Teaching of Chemistry" in Greystones, Ireland, February 29 to March 11, 1960. This seminar was attended by delegates from 18 European countries and the United States. The papers, discussions, reports of the working groups, and recommendations of the seminar have now been incorporated in an OEEC publication entitled "New Thinking in School Chemistry." Copies can be obtained free of charge from the OEEC Mission, Publications Department, 1346 Connecticut Avenue, N.W., Washington 6, D. C.

Anyone who has the impression that only American secondary school chemistry has problems—or that only Americans are doing any thinking about their solutions to these problems—should read this book.

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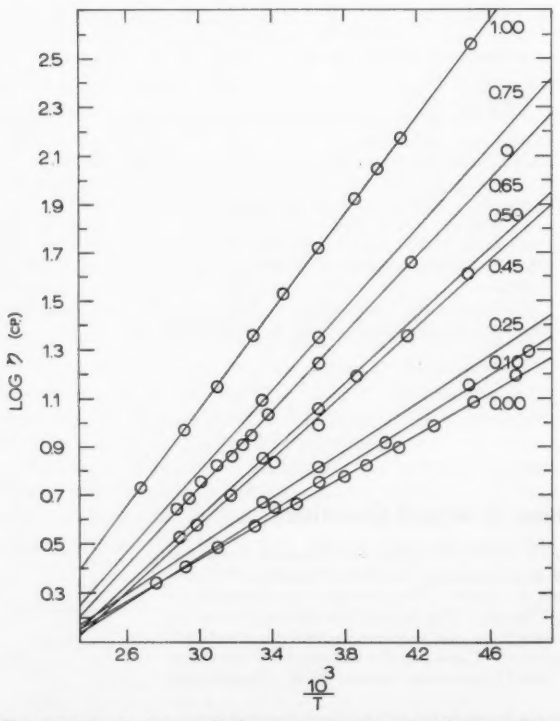
Viscosities of Butanol-Heptane Mixtures

A physical chemistry experiment

The Ostwald viscometer used in most physical chemistry courses is perhaps too simple a device. The usual attitudes of manual, student, and instructor toward an introductory relative viscosity experiment are perfunctory. We have attempted to make the experiment more memorable by using it to introduce the students to problems of temperature control below 0°C and to simple research in an old field. Since only equipment available to most physical chemistry classes has been used, the student experiment, as well as the new results, may be of general interest.

The work is begun by discussing with the students the qualitative relationship of liquid viscosity to liquid structure. It is emphasized that the most useful general relation, $\eta = Ae^{B/T}$, for the viscosity, η, of simple liquids holds within the accuracy of most experiments from temperatures slightly above the melting points to those slightly below the respective boiling points (*1*). The absolute rate theory interpretation of this result is discussed briefly (*2*). The students are then asked to make their own estimates of what common liquid in each of the classes, polar and non-polar, would offer the most critical test of the above equation; that is, what material is liquid over the largest range of temperature. *n*-Propyl alcohol (range, 220°) and *n*-heptane (190°) are accepted as reasonable answers.



The viscosities of mixtures of *n*-heptane and *n*-butanol as functions of the absolute temperature. The mole fraction of butanol is indicated at the upper end of each line.

The students find in the literature that viscosities of propanol and heptane seem to be readily available over temperature ranges of only 70° and 163°, respectively (*3*, *4*). *n*-Butyl alcohol is therefore substituted for propanol and data from −51 to 100°C (*3*) are found to be well represented by the equation η (cp) = 0.0011 × exp (2306/T). Data for heptane taken at temperatures from −73 to 90°C are almost as well represented by η(cp) = 0.178 exp (989/T). It is noted that, unlike most constants of rate processes, the constants of these equations have been determined over large temperature ranges, of order 1.0 T to 1.8 T(200–360°K), so that inaccuracies should be relatively small. It is stressed that the larger coefficient in the exponent for butanol is typical of hydrogen-bonding substances.

The problem of representing the viscosities of liquid mixtures is then introduced (*2*, *5–7*). From the foregoing it appears that the most concise and fundamental presentation of data would be the relation of the mole fraction, X, of the mixture to the two constants, A and B, of the above equation. The probable reason that this course seldom has been followed is low accuracy of the values of A and B which can be derived from existing data. The constants usually are determined over temperature ranges of order 1.0 T to 1.1 T and the largest range presently available seems to be 1.0 T to 1.5 T (*8*). It is proposed that it would be worth while to study mixtures of two liquids for which these ranges could be increased appreciably. One could then hope to interpret A and B as functions of X.

The students are reminded that there will not be many liquid pairs which will remain completely miscible while the absolute temperature is reduced by almost one-half. Butanol and heptane may be such a pair and would be most attractive because of the data already discussed for the pure substances and because reagent grade material is inexpensive in each case. However, a literature study shows only that the two liquids form an azeotropic mixture (bp minimum of 93°C) at 18 mole % butanol (*9*) and that some heats of mixing have been measured at temperatures as low as 8°C (*10*). It is therefore necessary for the students to test mixtures. These tests indicate complete miscibility between −80 and 90°C.

Consultation of a reference work for research (*11*) shows that the Ostwald viscometer is suitable for viscosities between 0.22 cp (heptane at +90°C) and 36.1 cp (butanol at −51°C) and makes clear the simple

precautions necessary for its use at room temperatures and above. Problems of adaptation of the Ostwald viscometer to low temperatures are considered. An unsilvered Dewar flask is taped (except for an observation port) for use as a thermostat. A toluol thermometer graduated in tenths of degrees and a solid CO_2-acetone mixture complete the equipment for temperature control. To avoid condensation of atmospheric moisture in the viscometer, a $CaCl_2$ tube is attached to each arm. To check the effect of a large temperature change on the standardization of the viscometer, pure heptane is used at −70°C and at other temperatures if necessary. The density of each mixture at each temperature is measured with a Westphal balance.

We find that the student measurements proceed smoothly at both low and high temperatures. Commercial reagent grade materials obtained over a period of three years reproduce the literature values of the viscosities of the pure liquids at 20°C. Each pair of students prepares one solution, studies it over the temperature range −65 to +70°C (1.0 T to 1.7 T), and determines values of A and B of the above equation such that the data obtained are represented within experimental error, that is within 2–5%.

As the students prepare to summarize their results, their attention is directed to the many proposed methods of analysis. Viscosities of liquid mixtures have been discussed from the purely empirical view (*7*), from the semi-empirical view (*2, 5, 6*), and from the simple theoretical view (*6*). The second view is useful for interpreting the A and B values determined above. A more sophisticated approach cannot be justified for a hydrogen-bonding liquid. Student values of A and B determined from the accompanying figure are presented as functions of the mole fraction of butanol in the table. A decreases sharply as X increases. B increases with X, slowly at first and then more rapidly, and in a third stage from about $X = 0.6$ to $X = 1.0$, follows the linear interpolation between the values of the pure liquids.

Constants of the Equation $\eta = Ae^{B/T}$ for Mixtures of n-C_4H_9OH and n-C_7H_{16}

$X(C_4H_9OH)$	A(cp)	B(°K)
0.00	0.178	989
0.10	0.108	1083
0.25	0.0089	1117
0.45	0.0032	1550
0.50	0.0031	1557
0.65	0.0021	1840
0.75	0.0015	1970
1.00	0.0011	2306

In the absolute rate theory B is approximately proportional to the heat of activation for flow. Much of the difference of the B's of pure heptane and pure butanol is expected to be accounted for by the necessity of breaking the hydrogen bonds of associated butanol to form the activated state. From the present results, one would conclude that, when butanol is diluted with heptane to X below 0.4, the heat of activation drops sharply because no hydrogen bonds need to be broken before a molecule can move relative to its surroundings. This interpretation is consistent with the values of A in the table and with the formation of an azeotropic mixture at $X = 0.18$. It may be noted that heats of mixing of the two liquids led independently to the conclusion that the double molecules of pure butanol are entirely dissociated in dilute solution (*10*).

The success of the simple approach outlined here shows that the undergraduate laboratory student may reasonably expect some of his afternoons to lead to significant new results. This extension of the effective temperature range for viscosities of mixtures easily could be doubled if one wished to introduce the student to the use of liquid nitrogen and organic solvents for temperature control between 143 and 210°K.

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Register of Scientists Interested in Overseas Assignments

In anticipation of the growing need for personnel under international programs in which it cooperates, the NAS-NRC is compiling a register of American scientists and other specialists who are interested in the possibilities of assignments abroad for periods ranging from several weeks to two years. Assignments become available irregularly throughout the year; they vary greatly in location, duration, stipends, and responsibilities.

Persons who wish to be considered for any such assignments are asked to fill out a special form, available upon request from the Committee on International Exchange of Persons, 2101 Constitution Avenue, N. W., Washington 25, D. C. Completion and return of the form will not constitute an application, but will ensure consideration for openings.

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A Controlled Atmosphere Thermobalance

The rapid growth of interest in the technique of thermogravimetric analysis (TGA) during the past ten years has generated a need for automatic recording thermobalances which will determine the pyrolysis of compounds either in a vacuum or in a controlled atmosphere such as argon, nitrogen, carbon dioxide, or hydrogen. A number of thermobalances, both manually operated and automatic recording instruments, which will operate under the above conditions, have been described in the literature.

A large number of instruments have been described which are based on the change in elongation of a quartz or metal spiral (1–5), similar to that first described by McBain and Bakr (*6*) in 1926. In many cases, an appropriate transducer element has been added such as a magnet and solenoid (*7–9*) a photocell and light beam arrangement (*10, 11*), a linear voltage differential transformer (*12*), or a mirror and light beam with a photographic recorder (*13*). Besides spiral type instruments, a number of torsion wire balances have been described (*14–17*) which will operate in a controlled gaseous atmosphere or in a vacuum.

One of the inherent problems of the quartz spiral type of balances is the fragility and limited load capacity of the spiral. To overcome this difficulty, an automatic recording thermobalance was constructed having a strain gauge as the weight-sensing transducer. A thermobalance has previously been described (*18*) employing a conventional analytical balance to which was attached, as the weight-sensing device, a length of strain gauge wire. However, the instrument could not be operated under controlled atmosphere conditions.

The strain gauge thermobalance is illustrated in Figure 1. The strain gauge used was obtained from Statham Instruments, Inc., Hato Rey, Puerto Rico, Model No. GIOB–O.15–350. The transducer has a range of ±0.15 oz with a sensitivity of 20.93 mv per oz at 9 v excitation voltage. Other models are available up to ±5 oz if a larger capacity instrument is desired. The strain gauge is a rather robust device in that it can stand a 1 lb force overload without damage. The excitation voltage can be direct or alternating current and it has a non-linearity plus hysteresis effect of less than ±1% of full scale. The gauge is small, 1.50 in. long, and weighs approximately 1.5 oz.

The strain gauge was mounted on an aluminum block attached to the aluminum cylinder housing by two small setscrews. The aluminum cylinder was 2.5 in. in diameter by 6 in. long and was machined from a solid metal block. One end of the cylinder was enclosed with a 0.25-in. thick plastic disk so that the four bridge leads could be connected to the outside power supply and recorder. The plastic disk was attached to the metal cylinder by six screws, along with an appropriate gasket and sealing compound. The glass system was attached to the aluminum cylinder by three setscrews and then sealed with Apiezon "W" cement.

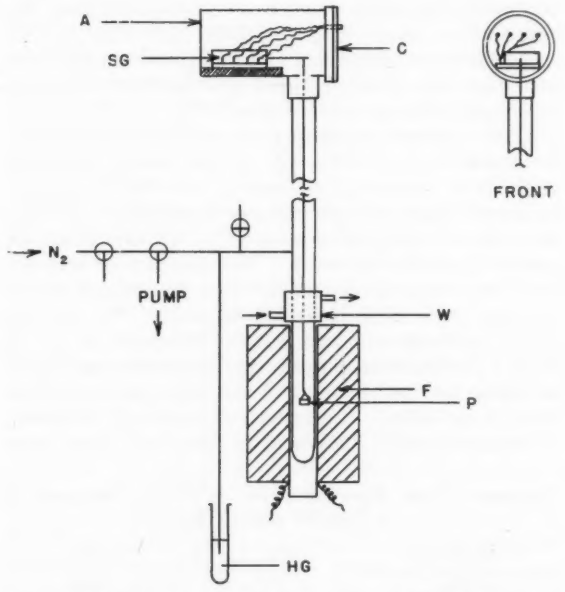

Figure 1. Schematic drawing of strain gauge thermobalance. A, aluminum cylinder housing; SG, strain gauge transducer; C, plastic disc; W, water-cooled standard taper joint; F, furnace; and P, sample pan.

To increase the sensitivity of the strain gauge, a length of aluminum metal, 0.25 in. × 1.25 in., was attached to the strain gauge probe. A platinum wire and sample pan was connected to the extension probe and allowed to hang freely into the glass system and furnace. The furnace chamber consisted of a 2.5-cm diameter Vycor glass tube which was connected to the Pyrex glass system through a water-cooled standard taper joint at *W*.

The bridge circuit for the strain gauge is given in Figure 2 and is a modification of the original circuit described by the manufacturer and also by Jackman (*19*). The power supply voltage must be quite stable, so two 6 v automobile-type lead storage batteries, connected in series, were employed. A 5KΩ Helipot was used to control the voltage into the bridge circuit. It should be mentioned that the bridge voltage must not exceed 9 v. An output filter circuit was necessary because of the pick-up of natural building vibrations

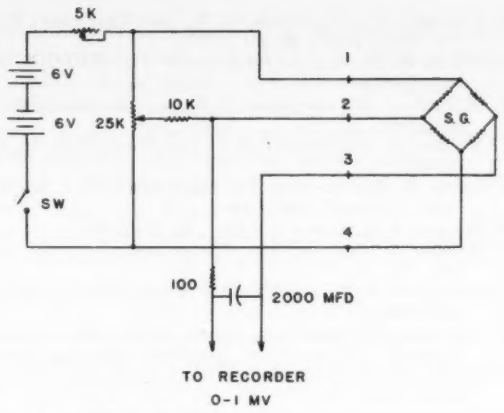

Figure 2. Schematic circuit for strain gauge.

and disturbances by the strain gauge when operating under this high sensitivity. The output from the bridge circuit was recorded on an E. H. Sargent and Co., Model SR, potentiometric recorder, using a 1.0 mv range plug and a chart speed of 0.1 in. per min. Full scale on the recorder corresponded to a change in weight of about 80 mg. However, other full-scale weight ranges could be obtained by decreasing the bridge circuit voltage by means of the 5KΩ Helipot.

The furnace consisted of a ceramic tube, 3.2 cm in diameter and 24 cm long, wound with No. 26 gauge Nichrome wire, and had a total resistance of 36 ohms. The furnace temperature was controlled by gradually increasing the input voltage by means of a motor-driven 0-135 v Powerstat (*20*). Maximum temperature of the furnace was about 850°C, although 500°C was the usual temperature limit employed. A typical furnace temperature-time curve is shown in Figure 3; the heating rate was 7°C per min. Also included in Figure 3 is a curve showing the change in weight of the pan as a function of temperature. In practice, a correction is made on the weight-loss curve to allow for this change in empty pan weight.

The operation of the thermobalance is illustrated by the thermogravimetric curves in Figure 4. The compounds studied were $CuSO_4 \cdot 5H_2O$ and $Na_2EDTA \cdot 2H_2O$ in nitrogen, and $Na_2EDTA \cdot 2H_2O$ in air. The thermal decomposition of $CuSO_4 \cdot 5H_2O$ should be independent of either air or nitrogen atmosphere; thermal decomposition of anhydrous Na_2EDTA, since it involves oxidation of carbonaceous material, should be dependent on the type of atmosphere present in the furnace.

The weight-losses involved for $CuSO_4 \cdot 5H_2O$ occurred at the following temperatures:

$$CuSO_4 \cdot 5H_2O(s) \rightarrow CuSO_4 \cdot 3H_2O(s) + 2H_2O(g) \ldots 45\text{–}100°C$$

$$CuSO_4 \cdot 3H_2O(s) \rightarrow CuSO_4 \cdot H_2O(s) + 2H_2O(g) \ldots 100\text{–}140°C$$

$$CuSO_4 \cdot H_2O(s) \rightarrow CuSO_4(s) + H_2O(g) \ldots 205\text{–}262°C$$

For the $Na_2EDTA \cdot 2H_2O$, the following weight-losses occurred, both in nitrogen and air:

$$Na_2EDTA \cdot 2H_2O(s) \rightarrow Na_2EDTA(s) + 2H_2O(g) \ldots 110\text{–}210°C$$

$$Na_2EDTA(s) \rightarrow Na_2CO_3 + \text{carbonaceous products} \ldots 230°C$$

The only differences between the two curves occurred above 300°C. Initial decomposition of the compound appeared to independent of the atmosphere employed and only at higher temeratures does this effect appear

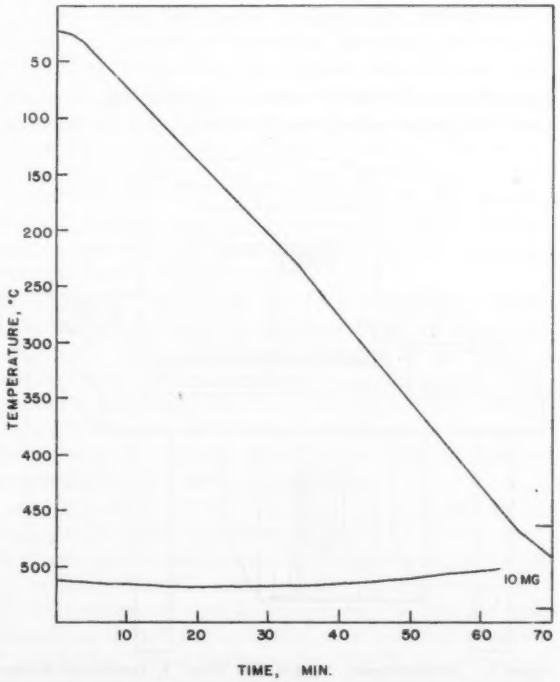

Figure 3. Furnace temperature-time calibration curve and change of empty pan weight with time.

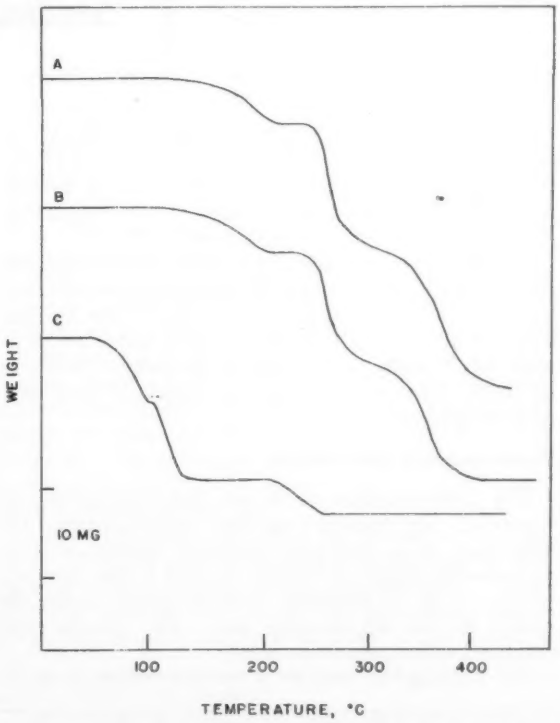

Figure 4. Weight-loss curves of various compounds. A, $Na_2EDTA \cdot 2H_2O$ (N_2); B, $Na_2EDTA \cdot 2H_2O$ (air); C, $CuSO_4 \cdot 5H_2O$ (N_2).

to be significant. By use of various atmospheres, it is possible to distinguish between the thermal pyrolysis of a compound and the resistance of the compound to oxidation from oxygen in the air.

From the weight-loss curves, the accuracy of the instrument on the weight axis was about ±1%, while on the temperature axis, about ±2%. The instrument has been in operation in this laboratory for the past several months and has performed as well as higher priced models. The entire thermobalance, excluding the recorder and labor costs, can be constructed for about $350.

The helpful assistance of Mr. Warner Kendall in construction of the balance is gratefully acknowledged. This work was supported in part by the U. S. Air Force, Office of Research and Development, through contract No. AF-49(638)-787.

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A Thermobalance for Student Experiments

Thermogravimetric analysis (TGA), an extremely valuable analytical technique, involves the weighing of substances while they are being heated. A complete treatment of the theory of thermogravimetry is available in the reference work by Duval.[1]

This paper presents the details of the construction and operation of an inexpensive, manually-operated thermobalance which was used for the study of the thermal stabilities of some metal chelates and which could be used for student experiments in thermogravimetry in advanced laboratory courses in analytical and inorganic chemistry.

Thermobalance Construction

The thermobalance is shown diagrammatically in Figure 1. In constructing the thermobalance, the glass base of an ordinary analytical balance was replaced with a base of bakelite so that a hole approximately 1/2 in. in diameter could be drilled under the center of the left balance pan. The balance was equipped with a magnetic damper.

For heating the sample, a furnace which could be heated to about 500°C was constructed from two concentric transite cylinders approximately 4 in. and 6 in.

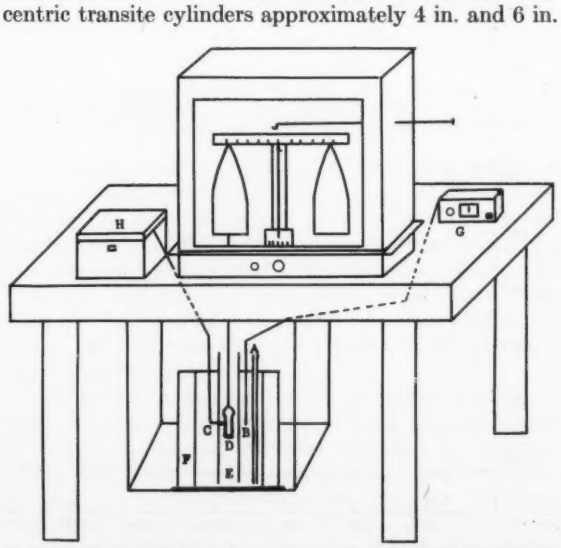

Figure 1. Thermobalance: *A*, gas inlet tube; *B*, temcometer thermocouple; *C*, iron-constantan thermocouple; *D*, sample vial; *E*, sample chamber; *F*, heating coils and insulation; *G*, temcometer; and *H*, potentiometer.

[1] Duval, C., "Inorganic Thermogravimetric Analysis," Elsevier Publishing Co., Inc., New York, 1953.

in diameter and 12 in. high. The heating element was a nichrome wire heating coil which was wrapped around the outside of the smaller cylinder and wired so that the total output was approximately 600 watts for the 110 volt input. The coils were covered with asbestos cement and the entire assembly placed inside the 6-in. cylinder. The two cylinders were attached to a 6-in. circular base cut from transite board. The space between the inner and outer cylinders was packed with mica insulation. The hot-junction of an iron-constantan thermocouple was sealed into the side of a piece of Pyrex tubing, 1 in. in diameter and 15 in. high. This tube was then put into the center of the smaller transite cylinder and secured using supports fashioned from triangular crucible supports made of nichrome wire. A space of approximately one-quarter inch was left between the bottom of the furnace and the Pyrex tube to permit the flow of nitrogen gas through the sample chamber. An inlet for nitrogen gas was provided by inserting a 15-in. length of 7-mm Pyrex tubing into the inner transite cylinder. The top of the furnace was covered with an asbestos mat, and the entire top area, except for the Pyrex tubes, was sealed with a layer of asbestos cement, about $^1/_2$ in. thick.

As shown in Figure 1, the balance and furnace were assembled on a small desk. The furnace was supported on a platform which was attached to the underside of the desk. The balance was secured to the top of the desk by putting the supporting legs into holes drilled into the desk top. In this way, any movement of the desk would not affect the alignment of the apparatus.

Care was taken to protect the balance from the heat of the furnace. The top of the desk was covered with transite board and the underside covered with asbestos paper. Glass wool was packed into the drawer of the desk (not shown in Figure 1) to provide additional insulation. To reduce air currents, the sides and back of the platform supporting the furnace and the sides and back of the desk were enclosed with asbestos paper.

The temperature of the furnace was controlled by a Temco input controller, Model CP-515T. The overall rate of heating was nearly linear, averaging 4–5° per minute. Temperature measurements of the sample chamber were made with a Leeds and Northrup double-range potentiometer indicator, Model 8657-C, using an iron-constantan thermocouple.

Small glass vials, 12 mm × 35 mm, with support loops made from 26-gauge nichrome wire, were used as sample containers. The vials were suspended in the furnace just opposite the iron-constantan thermocouple using a chain made from 20-gauge nichrome wire. Static heating tests showed that the vials could be heated to 450°C without showing any loss in weight; higher temperatures were not investigated.

A study of the effect of the furnace temperature on the balance using two different empty sample vials of known weight indicated that the balance was unaffected up to about 220°C. Above 220°, there was an apparent continuous loss in weight of the sample vial amounting to 1.7 mg at 450°C. A calibration curve of weight loss versus temperature (Fig. 2) was prepared from two such determinations and all data were corrected using this curve.

As a check on the accuracy of the final weight loss

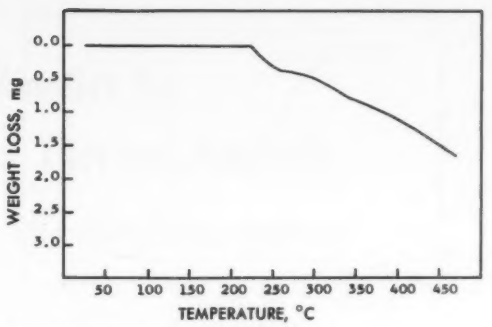

Figure 2. Thermobalance calibration curve.

readings obtained on the thermobalance, all sample containers were cooled in a desiccator after completion of a run and reweighed on a chainomatic balance. In every instance, the weights obtained were within 0.2 mg of the corrected weight loss as determined by the thermobalance.

Operation of Thermobalance

Before beginning a run, the furnace was flushed with dry nitrogen for a short while. A sample[2] of the compound to be studied was accurately weighed into a dry, weighed glass vial which was then suspended in the furnace. The balance was adjusted by first placing the 5-mg rider on the right-hand side of the beam over the 5.0 mg mark and sufficient weight was added to the right balance pan to bring the pointer to the desired rest point.[3] Nitrogen was passed through the sample chamber and the rate of flow, which could be approximated as the gas passed through a sulfuric acid bubbler, was regulated so it would not disturb the equilibrium of the balance.

The sensitivity of the balance[4] was then determined so that weight loss could be easily determined by noting the deflection of the pointer from its equilibrium position. After a weight loss of 2 mg, the rider was moved 2 mg to the left to restore the pointer to its equilibrium position. By successive reading of the pointer deflection and adjusting the rider to the left, when necessary, weight loss was easily determined as a function of temperature. Since both weight and temperature readings were taken together at definite time intervals, as measured by a stopwatch, the weight losses obtained were a function of time as well as temperature.

A typical thermogram obtained using the thermobalance described is shown in Figure 3 for the zinc-naphthazarin complex, $C_{10}H_4O_4Zn{\cdot}2H_2O$. The thermogravimetric data indicate a loss in weight of 12.3% at 232°C. The theoretical loss in weight for two moles of water is 12.4%. The dehydrated complex is apparently stable between 232° and 318°C.

It should be mentioned that the temperature range for the stability of a compound as determined from a thermogravimetric curve is only approximate. Newkirk[5] has recently pointed out that the establishment of the temperature range of stability of a compound

[2] The metal chelate samples were about 50 mg; much larger samples, however, may be used.

[3] This arrangement permitted a weight loss of up to 10 mg to be conveniently measured.

[4] The sensitivity was constant at 2.5 divisions per mg.

[5] Newkirk, Arthur E., *Anal. Chem.*, **32**, 1558 (1960).

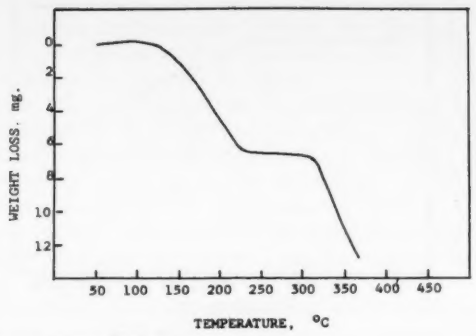

Figure 3. Zinc-naphthazarin thermogram.

is not simply a matter of reading the temperature at beginning and at the end of the level portion of a single thermogravimetric curve. The temperature will be approximately correct; it will, however, have been shifted upward in temperature by the effect of the rate of heating.

Although samples were run in a nitrogen atmosphere, other gases, such as oxygen, carbon dioxide, argon, etc., may be used. The thermobalance cannot be operated under vacuum conditions.

The cost of construction, excluding the analytical balance and potentiometer, was about $125; the most expensive item was the Temco input controller which cost about $85.

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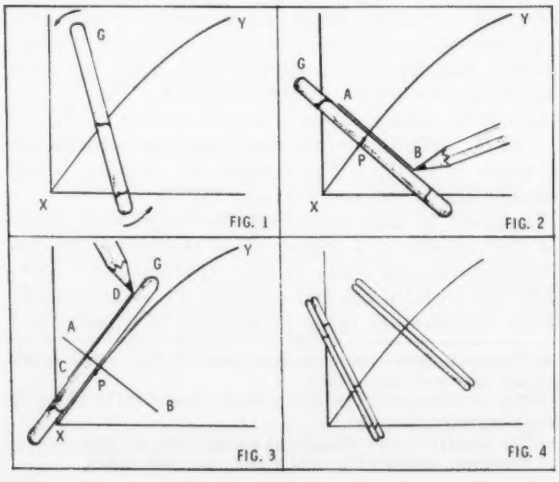
A Simple Method for Finding Slopes

It is the purpose of this paper to describe a simple method for determining quite reproducibly the tangent at any point on a given curve of any shape.

One proceeds as follows: A solid glass rod, G, $^1/_4$ in. in diameter and 6 in. long, is placed on a plot of a given curve, XY, as shown in Figure 1. The rod is rotated until an unbroken image of the curve is reflected through the glass rod as demonstrated in Figure 2. A straight line, AB, is drawn along and parallel to the axis of the glass rod. Next, as in Figure 3, the glass rod is placed across line AB so that again the line appears unbroken as it passes under the rod. As before, a line, CD, is drawn along and parallel to the axis of the glass rod. This line, CD, is the tangent to the curve at point P and is the slope of XY at point P. This procedure may be repeated for any point along XY to find the required slope at that point.

When a ray of light passes from one medium into another, its direction is changed and the magnitude of this change is a function of the angle of incidence.[1] If a plane of light strikes a rectangular prism normal to the surface of the prism, it will not be bent as it passes through and emerges from the other side because the angle of incidence is zero.



In the present case, instead of a rectangular prism, a glass rod is placed on a plot of the given curve. A plane of light perpendicular to both the plane of the paper and the axis of the glass rod will not experience any bending when passing through the rod. If the segment of XY covered by the rod is contained within such a plane, the light reflected from this line segment will not be bent and the curve will appear unbroken as it passes under the rod. A line, AB, drawn parallel to the axis of the rod, is perpendicular to this plane containing the line segment of XY and is therefore perpendicular to XY at P. In like manner, the line, CD, is perpendicular to AB and is, therefore, the tangent to XY at P. Since the intersection of a plane normal to a cylinder describes a circle, the reflection of the light from the segment of XY will produce a magnifying or lens effect.[2] This is why the broken images in Figure 2 have a sigmoid appearance which would not be observed if a rectangular prism were used. The attractive feature of this method is the use of an instrument (glass rod) readily available to the experimenter in any laboratory.

A somewhat more accurate method of determining CD is obtained by using two glass rods about $^1/_{16}$ in. in diameter and held parallel to one another as shown in Figure 4. This is true because the line segment covered by the $^1/_{16}$-in. rod is much less than that covered by the $^1/_4$-in. rod and, therefore, more closely approximates a straight line segment. Two rods are used because this procedure brings out to a greater extent small deviations from the perpendicular position.

[1] Hausmann, E., and Slack, E. P., "Physics," D. Van Nostrand Co., Inc., Princeton, N. J., 4th ed., 1957, p. 572.
[2] *Ibid.*, p. 607.

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Reaction Kinetics by Differential Thermal Analysis

A physical chemistry experiment

The determination of the reaction kinetics for homogeneous reactions is a common experiment in the undergraduate physical chemistry course. However, the study of heterogeneous reaction kinetics is a field of chemistry that is virtually untouched by standard physical chemistry courses. Outside of the initial cost of the equipment, the determination of the kinetics of a thermal decomposition reaction can be easily and simply carried out by the use of the technique of differential thermal analysis (DTA). The methods of DTA have been amply illustrated by Borchardt (*1*), mainly from the viewpoint of determining the heat of reaction from the dehydration of salt hydrates. Since the reaction kinetics can also be determined by this technique, this experiment is presented to supplement the previous heat of reaction studies (*1*).

There are a number of methods involving DTA that may be used to determine the kinetics of a thermal decomposition reaction. Most of them involve reactions of the type

$$\text{solid A} \xrightarrow{\text{heat}} \text{solid B} + \text{gas}$$

The first of these methods is that proposed by Kissinger (*2*, *3*), which was derived from the work of Murray and White (*4*) and Vaughn (*5*). This method is based on the equation.

$$\frac{d(\ln \phi/T_m^2)}{d(1/T_m)} = \frac{-E^*}{R} \tag{1}$$

where ϕ is dT/dt, the heating rate of the furnace, T_m is the peak temperature maxima, E^* is the activation energy, and R the gas constant. Once the above values are known, the frequency factor, A, can be calculated from

$$Ae^{E^*/-RT_m} = \frac{E^*}{RT_m^2}\phi \tag{2}$$

The above equations contain no terms relating to the reaction order, n. However, from the asymmetry of the curve (3), described by the shape index, S, the order of reaction, n, was shown to be equal to:

$$n = 1.26\, S^{1/2} \tag{3}$$

Thus, by measuring the peak maxima temperatures at a number of different furnace heating rates, and by plotting log ϕ/T_m^2 versus $1/T_m$, a straight line is obtained of slope $-E^*/2.3R$.

A second method, proposed by Borchardt and Daniels (*6*), was originally derived for the study of homogeneous kinetics but, as shall be shown later, can be applied to heterogeneous kinetics as well. This method is based upon the equation, derived for the first-order reaction ($n = 1$),

$$k = \frac{C_p(d\Delta T/dt) + K\Delta T}{K(A - a) + C_p\Delta T} \tag{4}$$

where k is the reaction rate constant, K, is a constant characteristic of the experimental apparatus, A is the total curve area, C_p is the total heat capacity of the reactant or reference material, ΔT is the differential temperature, t is the time, and a is the area under the curve up to time t.

The above equation was later simplified by Borchardt (*7*) to

$$k = -\frac{d(n/n_0)}{dt} = \frac{\Delta T}{A} \tag{5}$$

where k is the initial reaction rate, ΔT is the peak height (differential temperature), A is the total peak area, and $-d(n/n_0)/dt$ is the rate of reaction in fraction converted per unit time at the time (temperature) where the DTA peak height is ΔT. From a rough triangulation of the curve peak, $A = {}^1/_2\, \tau\Delta T_{max}$; where τ is the base of the triangle (peak) and ΔT_{max} is the height of the peak. Thus, it is seen that

$$k = -\frac{d(n/n_0)}{dt} = \frac{2\Delta T}{\tau\Delta T_{max}} \tag{6}$$

Another approximation of the Borchardt and Daniel's equation (*6*) has been derived (*8*, *9*). Since C_p is normally quite small, the equation simplifies to:

$$k = \frac{\Delta T}{A - a} \tag{7}$$

where the terms have the same notation as before. It is rather interesting to note that an identical method for determining k was developed by Allison in 1954 (*10*) from a consideration of the area under specific heat-temperature curves. The approximation method in equation (7) yields good results as confirmed by the thermal decomposition of metal oxalates. An excellent correlation was found between the DTA and thermogravimetric analysis (TGA) reaction kinetics data (*8*, *9*). Also, since this approximation method is simple to use, it is the basis for the determination of the reaction kinetics described in this experiment.

Experimental

There are many different types of DTA setups that may be used to determine the thermal decomposition curves. The apparatus described by Borchardt (*1*) may be used, if a small synchronous motor is attached to the Variac (*11*) so as to give the furnace a fairly linear heating rate. The apparatus described by Wendlandt

(*12*) may also be used. An inexpensive apparatus, developed over a period of years, is illustrated in Figures 1 and 2. The apparatus consists of a furnace and sample holder, a furnace temperature controller, a low-level dc amplifier (Honeywell, type 2HLA-7) and a strip-chart potentiometric recorder (Varian, type G-10). All of the above components are standard commercial items except the furnace and sample holder assembly, which can be easily constructed. Certainly other amplifiers, recorders, and furnaces may be substituted for the above components.

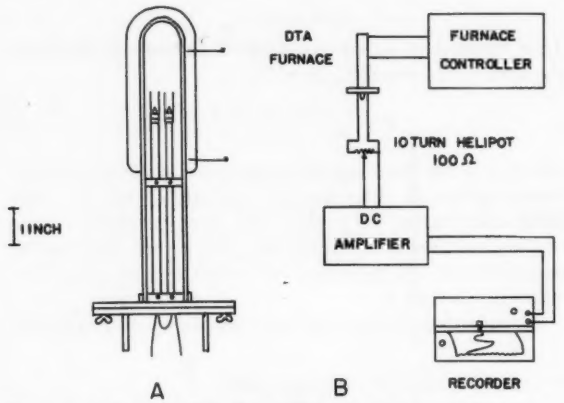

Figure 1. A, Schematic illustration of DTA furnace and sample holder. B, Schematic diagram of DTA apparatus.

The furnace consisted of an 8-in. length of 1-in. i.d. high temperature ceramic tube wound with enough Nichrome wire (1.06 ohms per ft) to give a total resistance of 25 ohms. The furnace windings were insulated with about a 0.5-in. layer of asbestos. The base of the furnace was constructed from 0.25-in. thick aluminum sheet in such a manner that the lower plate could be easily attached to the upper plate by two bolts and wing nuts. Attached to the bottom plate were two 0.25-in. o.d. diameter ceramic rods, each containing two 0.060-in. holes. The sample chambers consisted of two nickel or ceramic cylinders, 0.25 in. i.d. and 0.75 in. long, which are placed over the ceramic rod and thermocouple junctions. Each of the sample chambers could be easily removed for cleaning out the residue from the previously ignited sample.

The furnace temperature controller was constructed from two powerstats, type 116U O-135v, one of them driven by a 2 rph Cramer synchronous motor. The synchronous motor was attached to the powerstat through a 5:1 vernier radio tuning dial. The advantage of this system is that the tuning dial is inexpensive plus the fact that it acts as a friction clutch to allow the powerstat to be returned manually to its starting position.

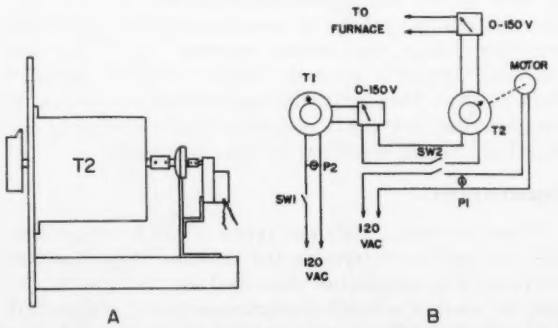

Figure 2. A, Schematic illustration of motor-driven powerstat. B, Wiring diagram for furnace temperature controller (schematic).

The $CaC_2O_4 \cdot 1H_2O$ used in this experiment was prepared by adding oxalic acid to a dilute calcium nitrate solution, digesting on a steam bath for one hour, and then filtering off the precipitate into a sintered porcelain crucible. After washing several times with water, the precipitate was allowed to dry in air for about 48 hours. The $NaHCO_3$ used was of reagent grade quality.

Procedure for the DTA run consisted of the following: 100 mg weight of sample; 10°C min^{-1} heating rate; previously ignited alumina being used as the reference material. Duplicate runs gave peak maxima temperatures reproducible to ±5%.

Results

The results of the DTA studies on $CaC_2O_4 \cdot 1H_2O$ and $NaHCO_3$ are given in Figure 3. Although only a a static air atmosphere can be maintained in the apparatus described herein, the $CaC_2O_4 \cdot 1H_2O$ was also run under helium in a controlled atmosphere DTA apparatus (*13*). The results obtained were quite different from those found in a static air atmosphere.

The thermal decomposition of $CaC_2O_4 \cdot 1H_2O$, as shown by thermogravimetric studies on an automatic recording thermobalance (*14*) and illustrated in Figure 4, takes place according to the following reactions:

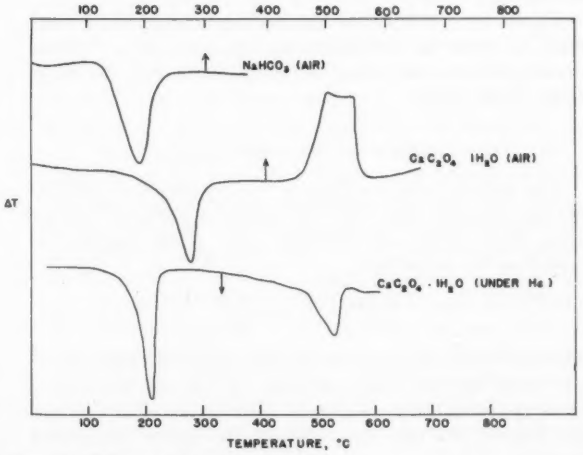

Figure 3. Differential thermal analysis curves.

$$CaC_2O_4 \cdot 1H_2O(s) \rightarrow CaC_2O_4(s) + H_2O(g)$$

and

$$CaC_2O_4(s) \rightarrow CaCO_3(s) + CO(g)$$

This thermal decomposition pattern is shown in the DTA curve (in air) by an endothermic peak for the dehydration reaction and a broad exothermic peak for the decomposition reaction. However, under helium, the second peak is an endothermic, rather than an exothermic peak. This difference can be explained in that the exothermic peak in the air DTA curve is the resultant of two competing reactions. One of them is the

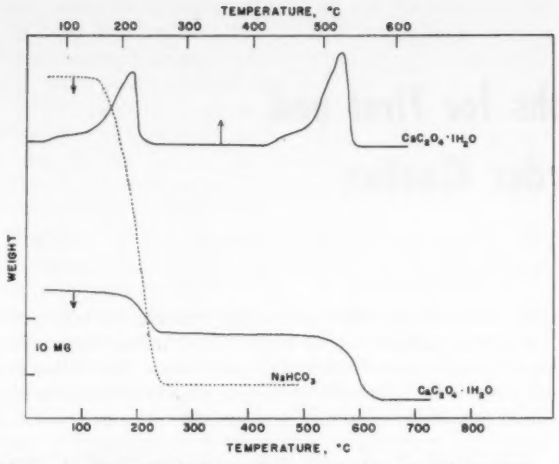

Figure. 4. Thermogravimetric analysis curves and a gas evolution curve.

above decomposition reaction while the second is the oxidation of carbon monoxide to carbon dioxide, according to the reaction

$$CO + {}^1/_2O_2 \rightarrow CO_2$$

which is a highly exothermic process. Thus, the effect of various atmospheres on the thermal decomposition reaction can be readily seen. Another illustration of the decomposition of $CaC_2O_4 \cdot 1H_2O$ can be seen in Figure 4 where the gas evolution is plotted as a function of furnace temperature. Since the thermal decomposition was carried out in helium (*13*), the first peak is due to water vapor, and the second peak is due to carbon monoxide evolution.

The kinetics of the dehydration reaction of $CaC_2O_4 \cdot 1H_2O$ was determined according to equation (7). Four points were chosen at random on the dehydration peak. The areas were obtained by integration of the peaks by the use of tracing paper and a semimicrobalance. The data obtained were in the following units, although other units may be employed: ΔT, in mm, while A and a were in mg of tracing paper. The value for E^* was obtained by use of the Arrhenius equation:

$$k = Ae^{-E^*/RT}$$

A plot of log k versus $1/(T)$ gave a straight line, as shown in Figure 5, of slope $-E^*/2.3R$. The value obtained for E^* in this study was 27 kcal mole^{-1}. A previous TGA method by Freeman and Carroll (*15*) gave 22 kcal mole^{-1}. The slight difference between results may be attributed to the effect of particle size, heating rate, furnace atmosphere conditions, and so on. By similar calculations, the kinetics of the decomposition of CaC_2O_4 (under helium) were also evaluated. An E^* of 85 kcal mole^{-1} was obtained, compared to a previous value (15) of 74 kcal mole^{-1}. However, the latter value was obtained in an air atmosphere.

To illustrate the above method with another compound, the thermal decomposition of $NaHCO_3$ was also studied. The decomposition reaction studied was:

$$2NaHCO_3(s) \rightarrow Na_2CO_3(s) + H_2O(g) + CO_2(g)$$

The DTA curve is shown in Figure 3 while the TGA curve is illustrated in Figure 4. From the reaction kinetics data obtained, E^* was found to be 20 kcal mole^{-1}. In this reaction, as well as in the above examples, the reactions were assumed to be first-order. The dehydration reaction of $CaC_2O_4 \cdot 1H_2O$ has previously been shown to be first-order also but the CaC_2O_4 decomposition reaction was 0.76 order (*15*).

The two examples listed above certainly do not exhaust the number of compounds that can be studied by this method. A series of hydrogen carbonates containing different cations may be studied as well as a number of inorganic salt hydrates. Many compounds evolve oxygen when heated as well as other gases such as carbon dioxide, sulfur dioxide, and sulfur trioxide; the list is virtually endless. It is hoped that this experiment will stimulate further studies in this exceedingly interesting area of investigation.

It is a pleasure to acknowledge the assistance of Mr. William Robinson and Miss Phyllis J. Kuhn.

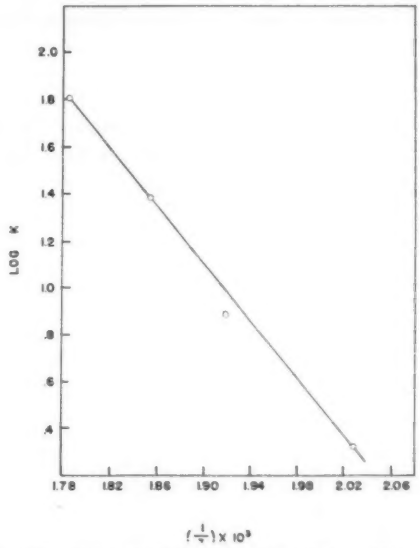

Figure 5. Arrhenius plot for the dehydration of $CaC_2O_4 \cdot 1H_2O$.

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Nomographs for First and Second Order Kinetics

In the study of reaction kinetics it is often necessary and useful to estimate rapidly the relation between a given per cent reaction, reaction time, and rate constant. For this purpose we have found the nomographs in Figures 1 and 2 more useful than the more general ones available in the literature.[3–5]

Figure 1 applies to reactions that follow first order kinetics; its use is readily seen from the following example. In the solvolysis of *p*-methoxyneophyl-*p*-toluenesulfonate in acetic acid at 50.0°, it was observed

Research supported by the National Science Foundation.

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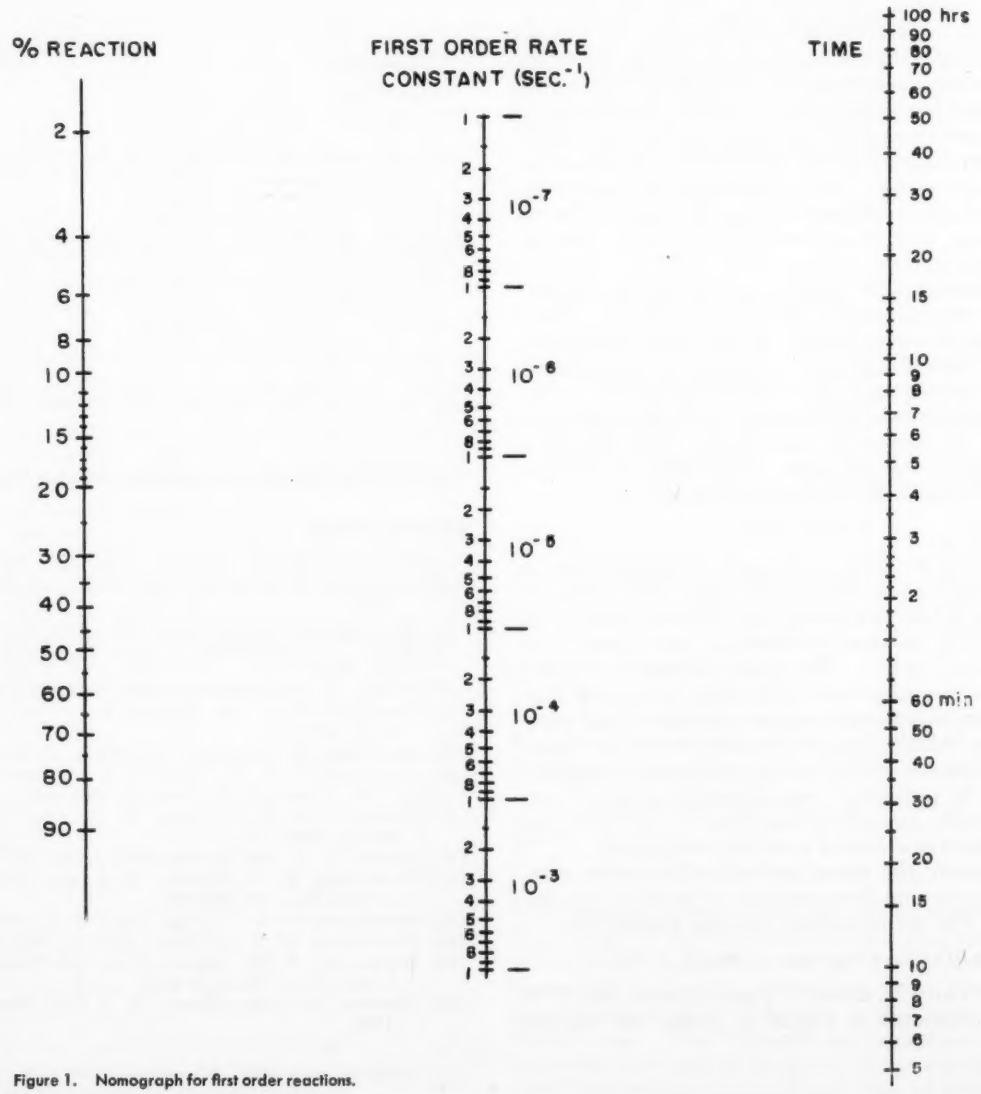

Figure 1. Nomograph for first order reactions.

that, after 15 minutes, 10.3% of the theoretical acid had been developed. By laying a straightedge across the nomograph joining these two points, one estimates a first order rate constant of 1.2×10^{-4} sec^{-1}. One is now in a position to program the pulling of samples to achieve a run of maximum utility. Thus for points at 25, 35, 45, 55, 65, 75, and 85% reaction one can estimate times 40 min, 1 hr, 1 hr 25 min, 1 hr 50 min, $2^1/_2$ hr, $3^1/_4$ hr, and $4^1/_2$ hr.

Figure 2 is for use with second order kinetics and corresponds to the two special cases of equimolecular reactants and reactants in 2 to 1 stoichiometric ratio. The numbers on the left hand side of the per cent reaction line are for use in reactions using equal concentrations of reactants, and those on the right hand side are for reactions in which one reactant is present in twice the concentration of the other. This second order nomograph is a little more difficult to use than the first order one in that two lines, intersecting on the center line, F(% Rx, Co), are required. For example, in the reaction of tetra-*n*-butylammonium bromide with 2-phenyl-1-propyl-*p*-toluenesulfonate in dry acetone at 75.0°, it was observed that, for initial concentrations of 0.04 and 0.02 M respectively, 17.5% of the inorganic bromide had been consumed after 15 minutes. A straightedge is laid on the nomograph connecting 17.5% reaction (on the 2:1 scale) and 2×10^{-2} on the initial concentration scale and intersecting the F (% Rx, Co) scale. A second straightedge is then used to connect this point to the observed time (15 minutes) and an estimate for k_2 of 6×10^{-3} liter/mole-sec is obtained. As pointed out above, the process can now be reversed so that aliquots may be taken at the optimum times.

For second order reactions with initial reactant ratios between 1:1 and 2:1, it is possible to interpolate between the two scales, though with larger errors (e.g., x% reaction for 1.5:1 falls roughly halfway between

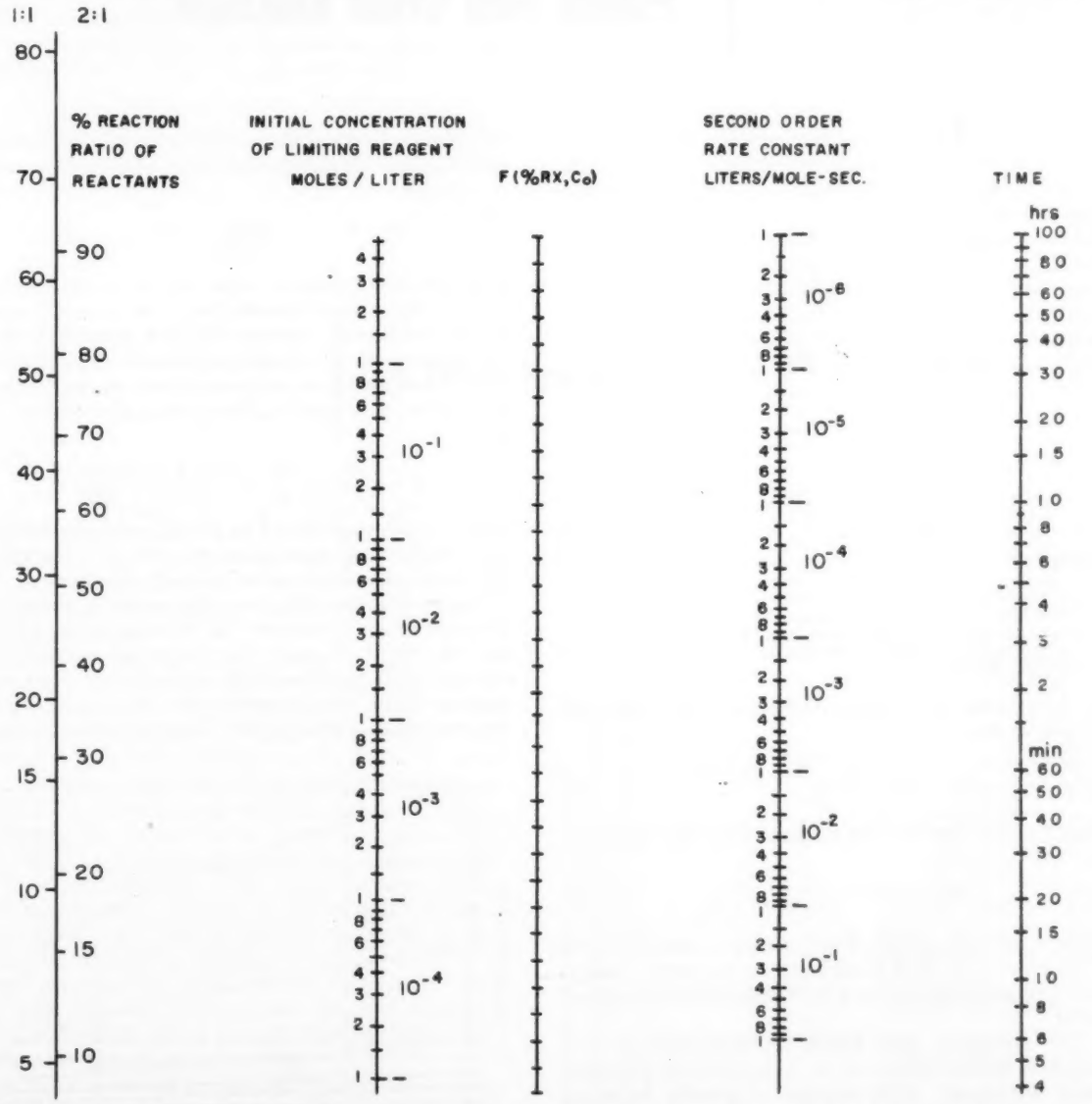

Fig 2. Nomograph for second order reactions. One line connects % reaction, initial concentration, and F(%Rx, Co). A second line connects F(%Rx, Co), the second order rate constant, and time.

the corresponding points for 1:1 and 2:1). For ratios much larger than 2:1, second order reactions follow first order kinetics to quite good approximations.

A further useful feature of both nomographs depends on the logarithmic nature of the time and rate constant scales; they can be easily extended. Thus times shorter than 6 minutes or longer than 100 hours may be employed by dividing or multiplying the numbers on the time scale by 10 (e.g., 10 hours becomes 1 hour or 100 hours) and correspondingly multiplying or dividing the numbers on the rate constant scale by 10 (e.g., 3×10^{-4} becomes 3×10^{-3} or 3×10^{-5}). For the second order nomograph, a similar relation holds between k_2 and initial concentration.

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Ratio Errors in Pseudo First Order Reactions

THE rates of second order reactions are frequently studied by pseudo first order kinetics. A "large excess" of one reactant is used, and the assumption is made that the concentration of this excess reactant remains constant throughout the reaction. Tacitly, an error is present due to the fact that the concentration of the excess reactant does not remain constant; only when the ratio of reactant concentrations is very large may second order reactions be validly studied by pseudo first order kinetics.

For the second order reaction

$$A + B \rightarrow X + \ldots\ldots \qquad (1)$$

the rate equation is

$$\frac{dx}{dt} = k(a - x)(b - x) \qquad (2)$$

where

a = initial concentration of reactant A
b = initial concentration of reactant B
x = instantaneous concentration of product X
k = second order specific reaction rate constant
t = time elapsed

In the general case, when a and b are not equal, (2) integrates to

$$k(a - b) = \frac{1}{t} \ln \frac{b}{(b - x)} \frac{(a - x)}{a} \qquad (3)$$

Equation (3) simplifies into a first order rate equation,

$$k(a - b) = k' = \frac{1}{t} \ln \frac{b}{(b - x)} \qquad (4)$$

when a is much greater than b, since x cannot exceed b, and $(a - x)/a$ is considered to be unity. When b is very small relative to a, k' is considered to be equal to ka.

The aberration that keeps (4) from being a true equation is the absence of $(a - x)/a$ in the log term, since the second order reaction is actually following equation (3) and the concentration terms are dependent variables. If equation (3) is divided by equation (4), the relation between the second order velocity constant and the observed pseudo first order velocity "constant," k', is found

$$\frac{k(a - b)}{k'} = 1 + \frac{\ln[(a - x)/a)}{\ln[b/(b - x)]} = 1 - E \qquad (5)$$

E always has a positive value and it is the fractional error in the observed pseudo first order constant caused by the assumption that $(a - x)/a$ is equal to unity in the derivation of equation (4). It is independent of the absolute values of a and b, and depends only on their ratio, a/b, and on the extent of reaction, x/b, since

$$E = \frac{\ln[(a - x)/a]}{\ln[b/(b - x)]} = \frac{\ln[(a/b - x/b)/(a/b)]}{\ln\,[1/(1 - x/b)]} \qquad (6)$$

Figure 1 was constructed by simply assigning values to a, b, and x, and calculating per cent E. Curves for any ratios of a and b can be similarly constructed.

Figure 1 shows that the ratio errors in the pseudo first order rate "constants" at the start of the reaction are 20, 10, 4, 2, and 1% for initial concentration ratios of 5, 10, 25, 50, and 100, respectively. That is, the product of the initial concentration ratio and the initial per cent error is always 100. The ratio error decreases

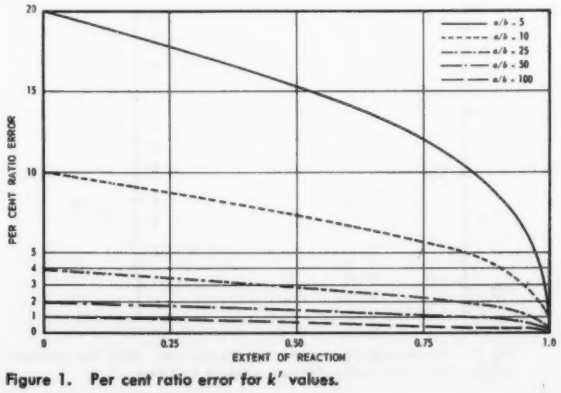

Figure 1. Per cent ratio error for k' values.

as the reaction progresses, since the ratio of the concentrations of the unreacted reactants is then increasing. As the reaction approaches completion, the error approaches zero.

If a second order reaction could be followed to completion, a plot of the instantaneous values of the pseudo first order velocity constants would have the general shape of one of the family of curves in Figure 1. Obviously, then, the graphical or averaged value of k' depends on the range in which the reaction is followed. The first part of a reaction is often stressed in kinetic studies, and unfortunately, it is during this part that the ratio error is greatest. During the latter stages of a reaction, the error decreases rapidly and becomes zero at 100% completion. The limiting value of k', that at the very end of the reaction, is not readily attainable by extrapolation of experimental k' values because of difficulties in experimental analyses when the concentration of B becomes very small and because of the rapidly changing slope of the k' curves during the late stages of reaction. However, this limiting value, which could be called a "true" pseudo first order velocity constant, may be calculated directly from any individual value of k' by use of Figure 1 or similarly constructed curves; i.e., by multiplying the observed value of k' by $(1 - E)$, the latter parenthetical term always being less than unity.

In kinetic studies involving solutions consisting of a small concentration of one reactant dissolved in another reactant acting as a solvent, the ratio errors discussed above are frequently negligible, and the pseudo first order rate constants by themselves have significance. Thus, even for a 1 M reactant in water, the maximum ratio error is only approximately two per cent, since the initial concentration ratio is about 55 to 1. In systems containing a third component as an inert solvent, however, the concentration of the excess reactant, and therefore, the initial reactant concentration ratio, may be so lowered that the maximum ratio error could greatly exceed the analytical errors. With such systems, Figure 1 may be used as a reference in choosing suitable ratios of reactant concentrations.

The authors wish to thank D. E. Pearson for pointing out the need for an analysis of this type.

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A Teflon Bearing for Stirrers

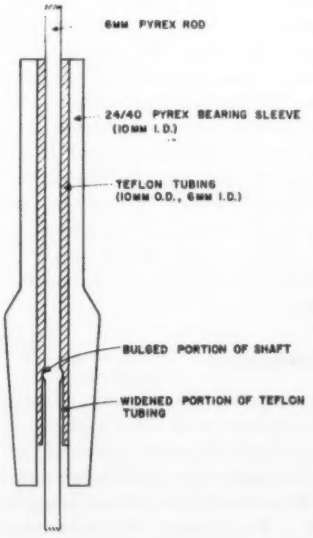
We have developed in our laboratories a stirrer bearing assembly which provides a good seal and has been used repeatedly at speeds of 3000–5000 rpm for periods of several days with no added lubrication. The apparatus may be constructed from readily available materials.

To make the bearing sleeve, Teflon tubing (10-mm o.d., 6-mm i.d.) is inserted in a Trubore type sleeve bearing (10-mm i.d.). The Teflon is not pushed to the bottom of the glass sleeve, to leave an area which will help keep liquids from creeping up the shaft. A tight fit is desirable so that rotation of the Teflon tubing is restricted. If there is some rotation, due to an imperfect match, the Teflon tubing may be wrapped with Teflon tape to increase effectively the diameter and thus to create a tight fit. The inner diameter of the Teflon tube is then widened at the bottom (approximately 8-mm i.d.) with a drill to a depth of approximately 20 mm (see drawing).

The stirrer shaft is made of 6-mm Pyrex rod. Because glass rods in a batch are not uniform, we select those pieces which give a snug fit and yet allow easy turning when placed in the bushing.

A bulge is fashioned in the rod by heating a small area and then pushing gently from both ends. The bulge should fit securely into the widened portion of the Teflon tube and seat up against the narrower portion.

To have a good seal during use, it is necessary to keep a slight pressure of the glass bulge against the Teflon seat. This can be accomplished by attaching the upper end of the stirrer shaft to a motor with a piece of rubber tubing. By fixing the motor in such a position that it keeps a slight tautness on the rubber tubing, the Teflon-glass seal will be maintained.


6MM PYREX ROD
24/40 PYREX BEARING SLEEVE (10MM I.D.)
TEFLON TUBING (10MM O.D., 6MM I.D.)
BULGED PORTION OF SHAFT
WIDENED PORTION OF TEFLON TUBING

A modification of this design for use in glassware without ground joints is possible. A glass tube placed in a rubber stopper may be substituted for the Trubore bearing sleeve. Teflon tubing is available in many diameters and thus different sizes of stirrer shafts can be made.

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Float Method for the Analysis of Solutions

The need often arises in chemical research to measure the concentration of solutions with a high degree of precision. If a sensitivity in the order of 1×10^{-5} grams/ml is required, ordinary methods are not suitable. Chemical or colorimetric methods will work when the total concentration is of this order of magnitude, but not at higher concentrations. Physical methods such as specific gravity or refractive index are not sensitive enough. An interferometer, often suitable for the application, is expensive, and the magnetically controlled float method (*1*) is complicated and rather expensive.

In this article we will describe a simple method of making precise and reproducible concentration measurements. This method, the thermal equilibrium float method, is not new, but deserves wider notice because of its simplicity and ease of operation. The only items of expense are a differential thermometer and a well-regulated water bath, and these are within the reach of every department.

In the thermal equilibrium float method, a float is suspended within the liquid whose concentration is to be measured. The temperature is adjusted until the float neither rises nor sinks. For a given float, a correlation can be made between this temperature and the concentration.

This method was first described by Richard and Shipley (*6*) in 1912. Lewis and McDonald (*5*) used this method for measuring the concentration of deuterium in water. They measured the density of water by finding the temperature at which a 10-cc float would neither rise nor sink in the sample, and obtained an accuracy in density of one part per million. Giffilan (*2*) used the same principle to measure the isotopic composition of sea water. He held the temperature constant at the ice-point and changed the buoyancy of the float by varying the hydrostatic pressure. The measurements were made in test tubes connected to a pump and manometer system. Hutchison and Johnston (*4*) applied the method to the accurate determination of the density of lithium fluoride by mixing two liquids of different density until the solid lithium fluoride would neither rise nor sink in the liquid mixture. Tollert (*7*) used the method to study the radial concentration gradient in laminar flow.

Theory and Method of Calibration

Consider a solid object suspended in a liquid, at such a temperature t_1 that the densities of solid and liquid are equal. Another solution of different concentration would cause the solid to be suspended at another temperature t_2, at which the density of the solution is again equal to that of the solid. To estimate the sensitivity of the method, it may be assumed that the density of the solid is independent of temperature. Then the concentration change corresponding to a small change in temperature depends on the change of density with concentration, $\Delta\rho/\Delta C$, and the change in density with temperature, $\Delta\rho/\Delta t$:

$$\Delta C/\Delta t = (\Delta\rho/\Delta t)/(\Delta\rho/\Delta C) \qquad (1)$$

A small value for $\Delta C/\Delta t$ is obtained if $\Delta\rho/\Delta t$ is small and $\Delta\rho/\Delta C$ large. Fortunately, this is the case for many systems.

Instead of measuring the individual factors in equation (1), however, it is more convenient to calibrate the float directly. A calibration curve is prepared by making up solutions of known concentrations, and measuring the equilibrium float temperature for each solution.

Description and Operation of the Apparatus

Key to the method is the float. This must be small so that temperature uniformity is achieved rapidly. Our floats are hollow spheres of soft glass, made by drawing a glass tube into a capillary, then blowing spheres in a tiny flame. This requires some practice, and it is usually necessary to make several floats before obtaining one of the desired density. A photograph of some floats, about 3 mm in diameter, is shown in Figure 1.

The remainder of the apparatus consists of a water bath with temperature regulated to 0.005°C, a Beckmann thermometer with 0.01°C graduations, and a test tube to contain the sample.

The sample of unknown solution is placed in a 20 ml test tube along with the calibrated float. The Beckmann thermometer is placed in the water bath. Experiment showed that the temperature in the test tube is not noticeably different from that in the water bath for the rates of heating and cooling used. The amount of solution taken is about 10 ml, so that the liquid level in the test tube does not touch the stopper when the

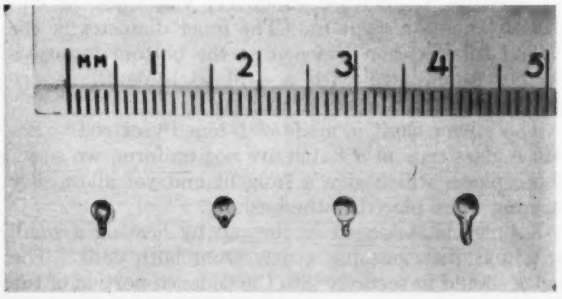

Figure 1. Photograph of floats.

tube is shaken to remove air bubbles from the float. To make a measurement, the temperature of the bath is increased slowly until the float starts to come down from the top of the liquid. At this instant the temperature control system of the bath is activated. By this time, the float has reached the bottom of the test tube. The temperature of the bath is now lowered until the float begins to move upward, at which point the temperature is changed by very small intervals until the float stays in one position.

Since the movement of the float is slow, it is usually possible to find the final temperature at which the float stays stationary in the liquid before the float reaches the top of the liquid.

Instead of viewing the float directly, it is more convenient to view the float by means of its shadow projected on a scale attached to the outside wall of the test tube. The surface of the float must be clean and free from air bubbles, which would result in an erroneous temperature reading. Air bubbles are dislodged by shaking and swirling the liquid in the test tube.

Results

To give an indication of the extreme sensitivity of the method, typical results for tap water and for distilled water are shown in Table 1, using the same float for both cases. This table shows typical observations of temperature and float motion made during the progress of a determination.

Table 1. Observed Float Motion for Tap Water and Distilled Water with Same Float

Time (min.)	Temperature reading	Direction of float motion
	Tap Water	
0	5.80	Down
6	5.53	Up
10	5.72	Down
14	5.57	Up
21	5.68	Down
25	5.63	Down
29	5.55	Stationary
	Distilled Water	
0	4.35	Down
5	4.15	Up
11	4.25	Up
15	4.29	Up
17	4.30	Down
20	4.295	Stationary

As shown in Table 1, the float motion is so sensitive that a change of a temperature of 0.01°C will cause a noticeable change of the float velocity. This high sensitivity means that small density or concentration differences can be detected.

To show how this method can be used for measurement of concentration, a series of solutions were made by adding weighed amounts of *n*-butyl alcohol to distilled water. The float equilibrium was measured for each solution. The corresponding temperature for float equilibrium is plotted against the weight percentage of *n*-butyl alcohol in Figure 2. Using a thermometer with a precision of 0.01°C, this method makes it possible to estimate the amount of *n*-butyl alcohol present in water within 0.001% with good reproducibility.

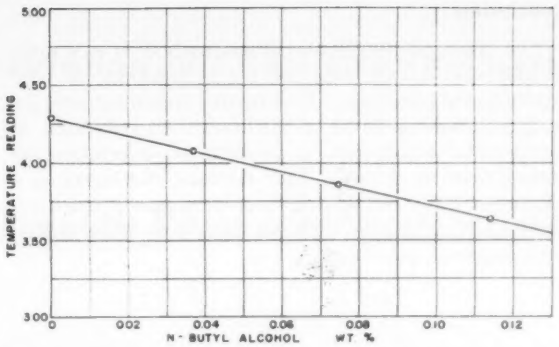

Figure 2. Equilibrium float temperature for different concentrations in the system, *n*-butyl alcohol and distilled water.

Comparison with Magnetic Float Method

The magnetic float apparatus is similar, but is a constant temperature device. The float contains a piece of iron, which is attracted by an electromagnet to keep the float suspended. The current through the electromagnet is correlated with density.

This method was compared with the thermal equilibrium float method for KCl solutions. The calibration curve prepared for the thermal equilibrium float by Goel (*3*) is shown in Figure 3. A temperature difference of 0.01°C corresponds to a concentration difference of 0.0004% KCl. In comparison, voltage and temperature fluctuations limited the accuracy of the magnetically controlled float to about 0.003% KCl. A concentration difference of less than 0.006% KCl could not be detected by refractive index measurements.

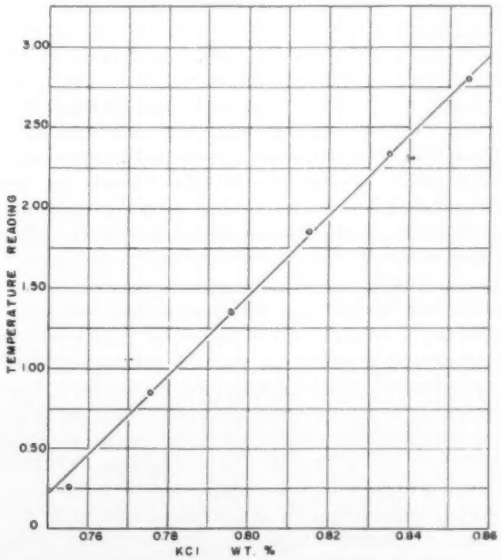

Figure 3. Equilibrium float temperature for different concentrations in the system, KCl and water.

The thermal equilibrium float method cannot be used when the temperature must be constant, as in the reaction rate measurements described by Cartan and Anacker (*1*).

Conclusion

The thermal equilibrium float method is a simple, reliable, and sensitive method for determining the concentration of solutions. It compares favorably with the magnetic float method in accuracy. A minimum of inexpensive equipment is needed to determine the temperature at which a float remains stationary in a solution. Since the float motion is extremely sensitive, small changes in concentration may be detected with a high degree of precision.

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Paper-made Crystal Models

Three-dimensional models of a number of simple ionic crystals can be easily constructed from a carton box of suitable size, pieces of carton paper and cellophane, adhesive tape and/or paste, and felt-point pens of blue and red colors.

The mode of construction of the simplest of them, i.e., the model of an NaCl-type crystal, is shown in Figures 1 and 2. Two large windows are cut in the front and back of the box, *A*, and a piece of carton paper, cut and bent, *B*, is fixed on the bottom of it by tape or paste. Five screens of cellophane with carton paper frames, *C*, are now prepared, and the positions of anions and cations on the <100> planes of the crystal are marked on the cellophane with felt-point pens in blue and red. All these frames are now inserted in the box, and their positions are adjusted and fixed by the hooks on both sides of each screen and by the cuts of *B*, with which the bottoms of the screens are held. Looking through the front window of the model set up in this way, a good stereoscopic view of the relative positions of ions in the true crystal can be obtained.

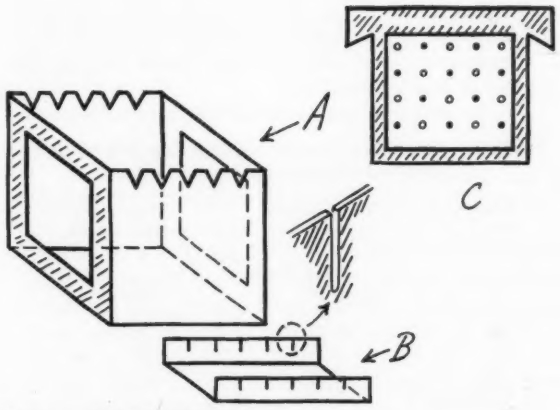

Figure 1. Parts of NaCl-type model.

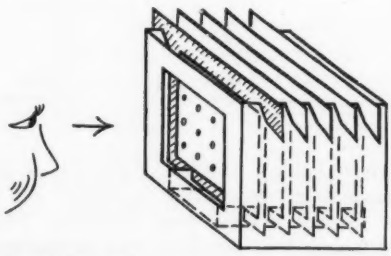

Figure 2. Assembled model.

Models of other types of ionic crystals, such as those of CsCl, zinc blende and fluorspar types, can also be constructed if special care is taken for their characteristic structural features. Even models of certain covalent compounds can be made; for example, a model of graphite can be obtained most easily, if the cellophane screens are identified with the planes of the hexagonal atomic "nets" in its crystal. In such a case, it is better to draw also the valence-bond lines on the cellophane, to make the structural features more easily perceptible. This is also true for the models of certain ionic crystals containing covalently bound particles, e.g., calcium carbide.

The merits of the present model can be summarized as follows: it is very cheap and easy to make, light and portable; it is inter-convertible, so that many structures can be demonstrated with one box, because the screens in it can be exchanged quickly; space can be saved to keep many models, because many screens can be kept together in one drawer, with only one box (or a few boxes) on the shelf. And, in some cases it also offers a possibility to take out certain important crystal planes (i.e., cellophane screens) from the crystal (i.e., box), and show them to the students separately, to give them a better understanding of the represented structure.

We call this model "the aquarium model," and sometimes "the marionette theater model," too, because many amusing scenes can be played in it.

Proceedings of the

California Association of Chemistry Teachers

John D. Roberts
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Pasadena

Nuclear Magnetic Resonance Spectroscopy

The use of nuclear magnetic resonance (NMR) spectroscopy in chemical research is spreading rapidly. Unfortunately, the cost and complexity of NMR spectrometers have largely precluded their use in undergraduate training. It is the purpose of this article to present the elements of NMR spectroscopy in a form which should be suitable for presentation to undergraduates and provide an introduction to more advanced treatments (*1–6*). Emphasis will be on absorption by ^1H and ^{19}F since these nuclei have so far been of the greatest importance in organic research. The discussion will be more illustrative than comprehensive.

Nuclear Magnetic Resonance Absorption

Many atomic nuclei act like charged spinning bodies, the circulation of charge producing a magnetic moment along the axis of rotation. Certain nuclei of particular interest to organic chemists—^1H, ^{13}C, ^{15}N, and ^{19}F—are classified as having spin I of $^1/_2$. This means that the magnitude of their magnetic moments in any given direction has only two equal, but opposite, observable values that correspond to spin quantum numbers equal to $+^1/_2$ and $-^1/_2$. Thus, if the nuclei are immersed in a magnetic field H taken as the z direction, they can only be regarded as being effectively lined up with the field ($I_z = -^1/_2$)[1] or against the field ($I_z = +^1/_2$). As with compass needles in the earth's magnetic field, the more favorable energy state is the one corresponding to alignment with the field. Of course, the difference in energy between the states ΔE is expected to be proportional to the strength of the applied field H at the nucleus and is equal to $\gamma\hbar H$, where $\hbar$ is Planck's constant h divided by 2π and γ is a proportionality constant typical of each variety of nucleus (^1H, ^{13}C, ^{15}N, etc.) (*1, 3*).

Obviously, we can in principle adjust the energy difference between the states with $I_z + ^1/_2$ and $-^1/_2$ to any desired value by suitably adjusting H; however, at ordinary temperatures, within the practically attainable range of values for H, the equilibrium concentrations of nuclei in the two possible states are always very nearly equal. This is because the energy differences between the states are quite small and because alignment of the nuclei with the field is opposed by thermal agitation in accord with Boltzmann's law (*1*).

In NMR spectroscopy, as with other kinds of spectroscopy involving absorption of electromagnetic radiation, the energy change ΔE associated with a given transition, as for a proton from $-^1/_2$ to $+^1/_2$ (Figure 1), is related to the frequency ν of the radiation by $\Delta E = h\nu$. Since $\Delta E = \gamma\hbar H$, we see that $\nu = \gamma H/2\pi$.

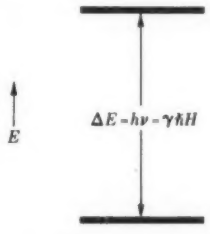


Figure 1. Energy levels for a single nucleus with a spin of $^1/_2$ in a magnetic field of strength H. Figures 1–8 are reproduced from "An Introduction to the Analysis of Spin-Spin Splitting in High-Resolution Nuclear Magnetic Resonance Spectra," W. A. Benjamin, Inc., New York, 1961, with the permission of the publisher.

To observe a nuclear resonance transition (assuming it is of the type classified as "allowed"), we have to adjust either the frequency ν of the radiation or the strength of the magnetic field at the nucleus H until $\nu = \gamma H/2\pi$. Experimentally, it is most convenient to hold ν constant and to vary H continuously with time until (and beyond) the point where resonance

The cover

Talent scouts for more than NMR experts may raid the CalTech campus. Senior Research Fellow Marjorie C. Caserio not only tames the multidialed beast in front of her, she is a wife, a mother, and a coauthor of an organic textbook.

"Cheesecake on the cover deserves pumpernickel on the inside." Even here the Pasadena variety is unique. Undergraduate Donald R. Davis, here shown with author Roberts, has been in full charge of the high resolution apparatus and published three papers as a sophomore.

The following description of nuclear magnetic resonance is in the main adapted from Chap. 1 of "An Introduction to the Analysis of Spin-Spin Splitting in High-Resolution Nuclear Magnetic Resonance Spectra," W. A. Benjamin, Inc., New York, 1961, with the permission of the publisher.

This is contribution No. 2722 from the Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena, supported in part by the Office of Naval Research.

Some of this material was presented at the Spring meeting of the ACS Student Affiliates of Southern California at San Diego State College. The lecture was a feature of the program at which winners of the annual undergraduate research paper competition were announced.

[1] The assignment of $I_z = ^1/_2$ for the more favorable energy state is used here, but not earlier (1), and corresponds to taking H in the negative z direction in conformity with Pople, Schneider, and Bernstein (3).

(energy absorption) occurs. In principle, resonance can be observed by a device as simple as the one shown in Figure 2. Here, ν is the frequency of an oscillator (customarily radio frequency *rf*), which bathes the sample in electromagnetic radiation, and H_0 is the strength of the applied magnetic field. When the field

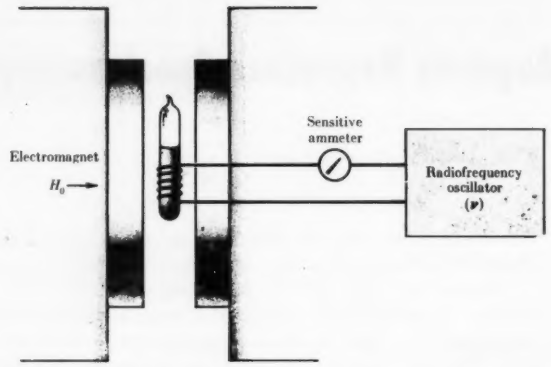


Figure 2. Schematic diagram of a simple nuclear magnetic resonance spectrometer.

at the nuclei H reaches the point where ν is equal or very close to $\gamma H/2\pi$, energy is absorbed. The current flow from the oscillator will then increase and can be detected by the ammeter in series with the oscillator coil. Further increase of H will eventually make $\nu << \gamma H/2\pi$, and the current will then decrease to its original value. In practice, the form of energy absorption curve as a function of H appears as shown in Figure 3, with peak centered on the point where $\nu = \gamma H/2\pi$.

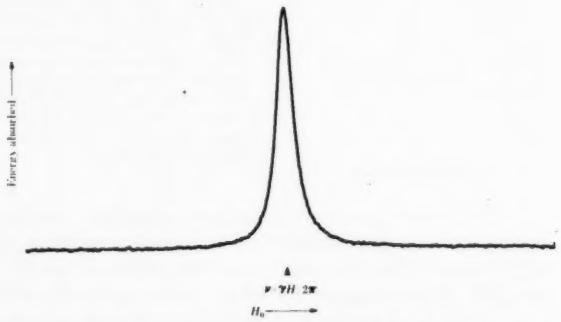


Figure 3. Typical energy absorption curve as a function of magnetic field *H*.

Chemical Shifts

Differences in absorption-line positions for nuclei of the same kind, such as protons or ^{19}F, but located in different molecular environments, are called chemical shifts (*7*). The magnitude of a given chemical shift is always taken in practice with reference to a standard. For protons, the customary standard was orginally the resonance-line position of the protons of water used as an external reference. Currently, tetramethylsilane is the favored standard substance; it is mixed with the sample to provide an internal proton reference line (*8*). Chemical shifts have their origin in diamagnetic shielding effects (*3*) produced by circulation of both bonding and nonbonding electrons in the neighborhood of the nuclei. These effects are strictly proportional to the magnitude of the applied field H_0 so that, for any H_0, the chemical shift $\delta_{AB}H_0$ between two nuclei A and B can be expressed by the following equation, where σ_A and σ_B are the shielding proportionality constants characteristic of each nuclei:

$$\delta_{AB}H_0 = \sigma_A H_0 - \sigma_B H_0 = (\sigma_A - \sigma_B)H_0$$

The chemical-shift parameter δ_{AB} is particularly useful for reporting differences in resonance-line positions because not all spectrometers operate at the same values of the magnetic field H_0. In practice it turns out to be very convenient to measure resonance-peak separations in units of cycles per second (cps). If frequency units are used and two peaks as for A and B are separated by n cps, then

$$\delta_{AB} = n/\nu$$

where ν is the oscillator frequency. Normally, δ values are computed with reference to a standard. Extensive tables of δ values relative to various standards are available for protons, ^{19}F, ^{13}C, ^{14}N, ^{17}O, etc., in a variety of typical molecules (*1, 3, 6*).

Figure 4 shows the NMR spectrum of diacetone alcohol at 60Mc with reference to tetramethylsilane as standard. The δ values corresponding to the line positions permit an almost unambiguous assignment of the groups of protons which give rise to each line.

For a given proton spectrum, the areas under resonance peaks can be found by electronic integration and are very closely proportional to the number of each kind of proton involved. Thus, the spectrum of ethyl alcohol shows three groups of peaks, which have areas in the ratio 1:2:3. These groups of peaks can be shown by isotopic substitution with deuterium (which gives no signal near that shown by protons), or by comparison of the spectrum with those of other alcohols to correspond to the resonances of the —OH, —CH_2—, and —CH_3 protons, respectively. Such measurements of relative peak areas have obvious utility in qualitative and quantitative analysis. With commercially available integrating equipment it is now possible to attain an accuracy of better than ±0.5%, in analysis either of total H or of the ratios of different kinds of H.

Comparisons of signal peak heights in place of comparisons of peak areas can give very erroneous results if the peaks do not have the same shapes. In general, resonance-line shapes are functions of many variables, and it is not uncommon for the peaks of a given sample to have quite different shapes (*9*).

Figure 4. Spectrum at 60 Mc of diacetone alcohol with tetramethylsilane as internal standard.

Spin-Spin Splitting—Qualitative Theory

Many organic molecules with protons on contiguous carbon atoms, such as ethyl derivatives CH_3—CH_2X ($X \neq H$), show principal groups of proton resonance lines as they correspond to chemical shifts; but each

group has a considerable fine structure that results from "spin-spin splitting" (*10*). Taking as a typical example the protons of an ethyl group as in ethyl iodide, the chemical shift difference between the methyl and methylene protons gives two groups of lines (see Fig. 5); these are split ("first-order" effect) into three and four lines, respectively, by spin-spin interactions. Several of the lines of the three-four pattern are further discernibly split as the result of what is called "second-order" spin-spin splitting. The chemical shift is easily recognized as such by the fact that the spacing between the main groups is directly proportional to H_0, the applied magnetic field. In contrast, the first-order splitting is field-invariant (at least when the chemical shift is large) and, for this reason, is easily recognizable also. The second-order splitting is field-dependent for rather complicated reasons (*3, 11*); it will not be discussed further here.

Figure 5. Nuclear resonance spectrum of ethyl iodide at 60 Mc.

The first-order splitting is independent of the applied field because it arises from fixed increments in the total magnetic field produced at a given nucleus by the magnetic moments of neighboring nuclei and transmitted by the bonding electrons (*1, 3*). Consider a nucleus undergoing resonance absorption that has a single neighboring proton. The effective magnetic field at the absorbing nucleus will have one of two possible values, depending upon whether or not the magnetic quantum number I_z of the adjacent proton is $+1/2$ or $-1/2$. The field of the proton thus either augments or decreases the applied field of the magnet. Since nuclear magnetic moments are independent of the applied field, the effect of the proton on the resonance absorption-line position will be independent of the applied field.

It is easy to demonstrate by isotopic substitution that the three-four pattern of lines for the spin-spin splitting of typical ethyl groups arises from the interaction of each group of protons with the other. Deuterons have much smaller magnetic moments than protons. Substitution of one deuteron on each methyl of an ethyl compound gives a triplet resonance for the methylene group (somewhat broadened because of the small magnetic effect of the deuteron), whereas substitution of two deuterons gives a doublet resonance with the splitting being due to the remaining proton. Thus, for this particularly simple case, the multiplicity of lines is $(n + 1)$ where n is the number of protons on the contiguous carbons. A number of similar examples are discussed elsewhere (*1–3*). That the methylene resonance of an ethyl group is not complicated beyond the observed quartet by the interaction of the magnetic effect of one of the methylene protons on the other is discussed in detail elsewhere (*11*). We only note here that chemically equivalent protons do not normally show spin-spin splitting. Thus, only single proton resonance lines are observed for H_2, CH_4, C_6H_6, etc.

In general, the effect of one proton on the resonance of another proton or group of equivalent protons depends on the number and kind of intervening chemical bonds and on the stereochemical relationships of the interacting groups. Normally, we expect splittings for nonequivalent protons in saturated systems to be approximately 15 cps when located on the same carbon, 5 to 8 cps when located on contiguous carbons, and essentially zero when separated by more than two carbons. Where restricted rotation or unsaturated groups are involved, widely divergent splittings are often observed (*1, 3, 6, 12, 13*).

The ratios of the line intensities in spin-spin splitting patterns usually follow simple rules (*14*) when the chemical shifts are large with respect to the splittings. A symmetrical doublet is produced by a single proton, a 1:2:1 triplet by two protons in a group, 1:3:3:1 quartet by three protons in a group, a 1:4:6:4:1 quintet by four protons in a group, etc. The intensities are seen to follow the binomial coefficients and this can be shown to be generally true for splittings caused by nuclei of spin $1/2$ (*15*).

The spectrum of acetaldehyde dimethyl acetal $(CH_3O)_2CHCH_3$ is shown in Figure 6 and provides an excellent example of how NMR shows the presence of contiguous protons. The symmetrical doublet and 1:3:3:1 quartet are typical of interaction between a single proton and an adjacent group of three. The methoxy protons are too far away from the others to give demonstrable spin-spin splitting.

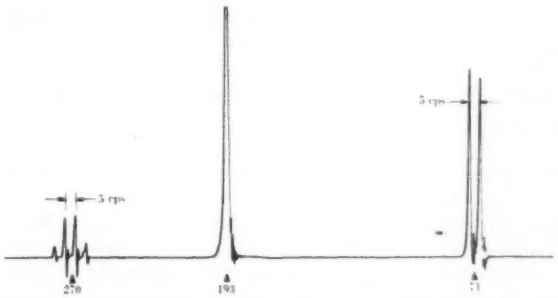


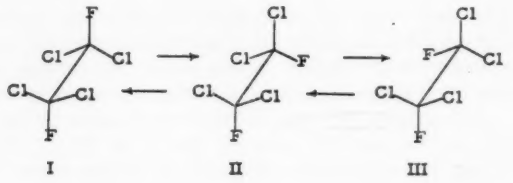
Figure 6. Spectrum of acetaldehyde dimethyl acetal at 60 Mc with tetramethylsilane as internal reference.

Nuclear Magnetic Resonance and Rate Processes

An NMR spectrometer is unusual among instruments used to study molecules through absorption of electromagnetic reaction in that it acts like a camera with a relatively long shutter speed. In fact, its shutter speed is very commonly about the same as a conventional camera having exposure times of a 100th of a second or so. When we take an NMR spectrum of a molecule which is undergoing any rapid motion or reaction, the result is something like taking a picture of a turning spoked wheel with a box camera. If the wheel turns only once a minute, a photograph at a 25th of a second will show the individual spokes without much blurring. On the other hand, if the wheel turns 100 or more times a second, then a photograph does not show the individual spokes at all but only the average outline of the rim and hub as a border of the gray of the spokes.

Pictures showing blurred individual spokes result only when the wheel is not turning either very rapidly or very slowly with respect to the camera shutter speed.

An extraordinarily vivid example is afforded by the fluorine (^{19}F) resonance spectrum of 1,2-difluorotetrachloroethane (*16*). This molecule may exist in three rotational conformations (I–III). Of these, II and III are optical isomers and should give identical NMR spectra. The chemical shifts of the fluorines of I are not expected to be exactly the same as of the rotational isomers II and III so that, in principle, one could observe separate NMR resonances for the isomers. However, at room temperature, rotation around the C—C bond occurs so rapidly that only a single fluorine resonance line is obtained, which is an average position as would depend on the proportions of time the molecules exist as I, II, and III. The rate of rotation about the C—C bond decreases with temperature and at −120° is so slow that one can now determine the separate resonances of I and the optical isomers II and III as shown in Figure 7. Studies of the blurring of the NMR spectrum of 1,2-difluorotetrachloroethane with temperature provide a means of evaluating the height of the rotational barrier.



Among the many further interesting examples (*1, 3, 4, 17, 18*) of the effect of rate processes on NMR spectra might be mentioned the allyl Grignard reagent, CH_2=CH—CH_2MgX (*19*). This substance in ethereal solution might be expected to show three vinyl proton resonances and a resonance typical of a CH_2 bonded to magnesium. Instead, besides the typical four-three pattern of the ethyl groups of the solvent, only a strong doublet four-proton and a quintet one-proton resonance are observed at 148 and 377 cps, respectively. Only the one-proton peak at 377 cps is in the region where vinyl protons usually absorb. The four-proton peak comes just where one would expect to find the *average* of two vinyl protons and the two protons of a —CH_2—MgX group. Apparently, the magnesium is shifting very rapidly back and forth between the two end carbons of the allyl group so that each is a CH_2= carbon and a

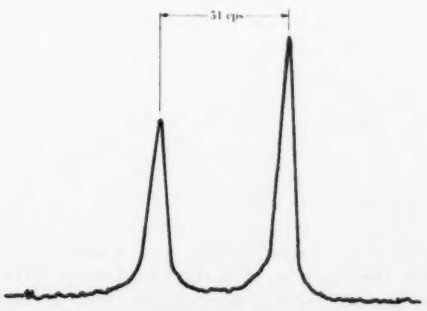


Figure 7. Fluorine spectrum of 1,2-difluorotetrachloroethane at −120° and 56.4 Mc.

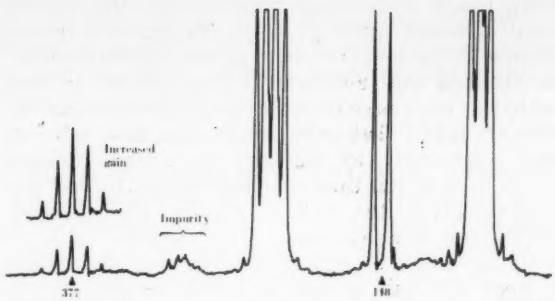


Figure 8. Proton spectrum of allylmagnesium bromide in diethyl ether with tetramethylsilane as internal standard. The strong triplet and quartet resonance (unindexed) arise from the solvent.

—CH_2—MgX carbon half of the time. The spin-spin splitting pattern agrees with this interpretation

$$CH_2{=}CH{-}CH_2MgX \rightleftarrows XMg{-}CH_2{-}CH{=}CH_2$$

Amazingly, this equilibrium still seems to be established more rapidly at −80° than the time of observation.

Nuclear magnetic resonance is radically changing the practice of organic chemistry. The advent of a routine analytical instrument (Varian A-60) augurs well. The operating principles of the spectrometer can be readily grasped by students with high school or freshman physics and, furthermore, very interesting and important NMR research can be carried out which does not require a knowledge beyond first-year chemistry.

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(15) Ref. (5), Chap. 10.
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Chem Ed

BOOK REVIEWS

Chemistry

Michell J. Sienko and *Robert A. Plane*, both of Cornell University, Ithaca, New York. 2nd ed. McGraw-Hill Book Co., Inc., New York, 1961. xiii + 623 pp. Figs. and tables. 17 × 24 cm. $7.50.

At first glance the second edition of this popular general chemistry text would seem to be little changed from the first. A more careful examination reveals that many significant, though not extensive changes have been made. For the most part they have corrected or strengthened the relatively few weak points of the first edition, resulting in improvement of an already high quality textbook.

Two of the changes, however, reverse this trend—the new print is not quite as boldly clear and easily read as formerly, and the fine print, which is much more frequently used, is now too small to read easily.

The book is attractively bound, and its general organization is unchanged. There are slight modifications in many of the figures, such as the use of gray and black tones of greater contrast than formerly and improved labeling where needed. A few new figures have been added. With very few exceptions, such as the over-simplified diagram representing the Millikan oil-drop experiment, the illustrations are excellent. Most, but not all of the numerical problem exercises have been altered enough to change the answers, and the total number of exercises has been increased by about ten to fifteen per cent.

An improved section entitled Electron Probability Distributions replaces the one called Wave Mechanics and appropriately introduces the concept of orbitals (Chapter 3). In the same chapter, size of atoms is now discussed in terms of atomic radii, rather than giving emphasis as formerly to the less reliable concept of gram-atomic-volume.

In Chapter 4, a section called Saturation of Valence remains unchanged. This is one of the less effective treatments. A good section on Shapes of Molecules has been added, however.

Except for the problems, the chapter on stoichiometry is essentially unchanged. The discussion of the difference between oxidation number and valence has been relegated to fine print, although it is certainly not of minor importance in view of the all-too-common tendency to use these terms interchangeably.

Other changes include: improvement of the presentation of the principle of diffraction of X-rays by crystals (Chapter 8); addition of a section, Entropy and Spontaneous Change, and improvement of the appearance of the phase diagrams (Chapter 9); omission of the former seemingly dislocated opening section, Multicomponent Systems, and the section entitled The Solution Process (Chapter 10); inclusion of the concentration term, *formality* (unfortunately confusing as presented); promotion of the section on solubility from the end to the middle of Chapter 10, resulting in improved order; expansion of the discussion of the Brønsted-Lowry theory in the same chapter; addition of a much needed section on buffer solutions (Chapter 17); and use of arrows instead of dots to represent the electrons in orbitals.

The changes in the second half of the book, which includes most of the descriptive chemistry of the elements and their compounds, are truly minor. The few changes made were dictated by good judgment. The alterations consist of slight changes in wording here and there, modifications of some figures, and the addition of a brief paragraph or so (in fine print) outlining the behavior of the elements in the conventional qualitative analysis scheme, where applicable. The discussions of the elements of relatively minor importance appear in fine print.

The book is well indexed, with every indication that the indexing was revised in keeping with the changes in content. Useful new features include a periodic table inside the back cover and a table of equilibrium constants added as an appendix. One may observe that both authors have been promoted one step on the professorial ladder of rank since the first edition. Doubtless this recognition was at least partially the result of their having authored a well-conceived, well-written and well-received textbook of general chemistry!

A new edition of the Instructor's Manual has been prepared to accompany both text and laboratory manual. The content of the sample examinations and quizzes has been completely revised. The greatest change is the addition of a seventeen-page unit giving directions and suggestions concerning the laboratory program. The Manual is well written and should be very helpful to the instructor.

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Reviewed in This Issue

Michell J. Sienko and *Robert A. Plane*, Chemistry
Herbert W. Cremer and *Sidney B. Watkins*, Editors, Chemical Engineering Practice. Volume 10: Ancillary Services
D. O. Jordan, The Chemistry of Nucleic Acids
Leonid V. Azároff, Introduction to Solids
Herbert Weisz, Microanalysis by the Ring Oven Technique
J. A. Edwards, Laboratory Management and Techniques
H. Zeiss, Editor, Organometallic Chemistry
L. Ambard and *S. Trautmann*, Ultrafiltration
J. E. Gowan and *T. S. Wheeler*, Name Index of Organic Reactions
The Science Study Series. Volumes 12 and 18.
E. H. Rodd, Editor, Chemistry of Carbon Compounds. Volume 4. Part C: Heterocyclic Compounds
Wayne Sorenson and *Tod W. Campbell*, Preparative Methods of Polymer Chemistry
I. M. Kolthoff and *Philip J. Elving*, Treatise on Analytical Chemistry. Part 1, Theory and Practice, Volume 2
I. M. Kolthoff and *Philip J. Elving*, Treatise on Analytical Chemistry. Part 2, Analytical Chemistry of the Elements, Volume 1
Erwin Chargaff and *J. N. Davidson*, Editors, The Nucleic Acids. Volume 3
G. Masing, Ternary Systems
Editors of Industrial and Engineering Chemistry, Modern Chemical Processes. Volume 6
Robert Brent, The Golden Book of Chemistry Experiments
Klare S. Markley, Editor, Fatty Acids: Their Chemistry, Properties, Production, and Uses. Part 1
Reagent Chemicals. American Chemical Society Specifications, 1960
Herbert R. Simonds, Source Book of the New Plastic, Volume 2

Chemical Engineering Practice. Volume 10: Ancillary Services

Edited by *Herbert W. Cremer* and *Sidney B. Watkins*. Academic Press Inc., New York, 1960. vi + 606 pp. Figs. and tables. 16.5 × 25 cm. $19.50.

Volume 10 contains a great deal of updated information on fuels, combustion systems, steam, power, water, and waste disposal.

The section on fuels covers solids (24 pages), liquids (25 pages), and gases (58 pages). The section on combustion systems for various fuels (53 pages) is very well illustrated and contains sub-sections on principles of combustion, mechanically fired systems, systems for low grade fuels, combustion of vegetable wastes, pulverized fuel firing, high intensity combustion systems, control methods, and a sub-section on types of burners.

A section on steam (114 pages) covers the various types of boilers and auxiliary equipment, automatic control and instrumentation, economy of steam raising, waste heat boilers, steam distribution problems, combined power and heat systems, and is also well illustrated.

The section on electrical installations (62 pages) is a good compilation of interesting and informative material for the design of electrical systems.

The section on water supplies (111 pages) contains a sub-section on water quality (76 pages). Material in this section is very similar to other material that has been published except that the presentation is well organized and placed in one compact volume. The last section on effluent treatment and disposal (133 pages) presents a good discussion on pollution problems which is general enough so that the information would be applicable in this country as well as in the European countries.

The writing of this volume is consistent with the excellent presentations in the previous volumes.

Volume 10 is published out of sequence. Volumes 1 to 6 and Volume 11 have been published. Further volumes are in preparation.

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The Chemistry of Nucleic Acids

D. O. Jordan, University of Adelaide, South Australia. Butterworths, Washington, D. C., 1960. ix + 358 pp. Figs. and tables. 15 × 22 cm. $10.50.

This book is devoted solely to the organic and physical chemistry of the nucleic acids and their components. Its best use would appear to be as a text for the rather rare chemistry course restricted to these topics, although it would also be of value to the physical or organic chemist wishing a relatively uncluttered, relatively inexpensive presentation of the chemistry of these biologically important compounds.

The treatment approaches that of a literature review, replete with references but deficient in critical evaluation. Most of the writing was apparently completed in 1958. A number of significant recent advances are therefore absent. One puzzling feature is the omission of any discussion of the methods developed by Khorana and his associates for the synthesis of nucleotides and oligonucleotides.

One must note that most of the material of Jordan's book can be found, usually in more detail, often in similar fashion, and in part supplied by Jordan, himself, in the three volume treatise, "The Nucleic Acids," edited by Chargaff and Davidson. A number of topics covered in the recent third volume of this work are somewhat more up-to-date than in the book under review. These volumes with their much wider range of topics must remain the best assembly of basic information on the nucleic acids. Few readers who have this set available to them will find Jordan's book to offer either significantly different information or a significantly different viewpoint.

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Introduction to Solids

Leonid V. Azároff, Illinois Institute of Technology, Chicago. McGraw-Hill Book Co., Inc., New York, 1960. xiv + 460 pp. Figs. and tables. 16 × 23.5 cm. $9.50.

This new text on the solid state should be sub-titled "from - a crystallographic point of view." The author's purpose "to use the crystallinity of solids as a framework in discussing their nature and properties" has certainly been fulfilled, but at the expense of discussing fabrication and evaluation methods, end uses of materials, or comparative properties. Thus while the book probably serves the purpose for which it was intended—the exposition of fundamental concepts of the solid state to students of varied backgrounds—it is not a source of specialized data in particular areas. Specialists will probably feel that their pet fields have been slighted. Chemists and physicists will notice the absence of such keywords as *crystal field, Jahn-Teller ion, pore conduction, bismuth telluride, garnet,* and *maser* in the Index or the text. Ceramists will probably be disappointed by the almost complete neglect of polycrystalline systems. Mineralogists will find nothing on high pressure phases such as coesite or jade. However, metallurgists should feel at home because much of the crystallographic discussion uses metallurgical examples.

The approach is non-mathematical and descriptive. Much of the mathematics which is used is not essential to the discussion and seems to be thrown in as window dressing. Each chapter has a set of exercises which greatly enhances the value of the book. Students should find these problems stimulating. Direct references to the literature are not used, but each chapter has a supplementary reading list, mostly on standard books in the field. There are only three 1958 and two 1959 references, giving a certain historical air to a subject which is experiencing a vigorous growth. On the other hand, the treatment is quite basic and sound and the book will be useful in years to come as a reference in such topics as atomic packing principles in solids. The text is somewhat austere and could be improved by the more frequent use of specific examples related to the principles being discussed. The austerity is heightened by the lack of tabular experimental data on typical compounds.

There is more material then can be used in a one semester course. The chapter layout will allow the instructor considerable leeway in the choice of subject matter.

The four appendices are useful, particularly the ones on Ahrens atomic radii and the listing of the 230 space groups according to the order in the International Tables. The Ahrens radii have proven very useful in studying transition metal compounds and their use should be encouraged.

A few typographical errors will not cause any trouble. There are some questionable statements such as saying that the spectral symbol *f* stands for "further" (it stands for "fundamental"), and saying that the peaks of permittivity vs. T and of C_p vs. T for $BaTiO_3$ occur at the Curie point for the same reason (permittivity decreases below the Curie point because of saturation effects).

All in all, the text will serve the intended purpose of elementary instruction, but there is still a need for texts in particular areas such as solid-state inorganic chemistry.

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Microanalysis by the Ring Oven Technique

Herbert Weisz, Technische Hochschule, Vienna. Pergamon Press, Inc., New York, 1961. 112 pp. Figs. and tables. 15 × 22 cm. $5.

The ring oven technique was introduced by Professor Weisz in 1954 as a means of accomplishing systematic separations on a single drop of solution. Since its introduction, the technique has been adapted to quantitative procedures and dozens of fascinating applications have been evolved, extending from the teaching of qualitative analysis to the handling of radioactive substances. The remarkable simplicity and utility of the ring oven technique has elicited widespread interest, and it is timely and fitting that Professor Weisz has collected the information on this method and made it available in the form of a monograph.

The scope of the book is sufficient to give enough information so even the non-initiated analyst can develop the necessary technique and find worth-while applications for the method. The discussion of the apparatus and its use includes a description of the ring oven, which is now commercially available, as well as the attendant bits of equipment for handling the small amounts of liquids and gases employed in ring oven operations. The description of working technique includes qualitative analysis without separations, as well as analytical schemes involving two and more groups. Systematic separation

schemes are given, and test reactions recommended for metal and acid ions are clearly outlined.

Many practical applications are suggested, and a complete list of pertinent references implement the use of the book. One of the remarkable results of the development of ring oven methods is the application in semiquantitative analysis, whereby spot tests are not only enhanced in sensitivity, but are made elegantly useful for spot colorimetry.

The book is strongly recommended for the practising analyst and for the teacher of analytical chemistry. The warm recommendations of both the technique and the book by Professor Fritz Feigl serves as a good indication of the merit of this contribution.

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Laboratory Management and Techniques

J. A. Edwards, Birmingham University, England. Butterworth, Inc., Washington, D. C., 1960. xvi + 207 pp. Figs. and tables. 14.5 × 22 cm. $7.

The largest and most important part of this compact volume is that directly suggested by the title, covering equipment, organization, operational schemes, precautions and limitations in academic chemical laboratory management. The reviewer was even a wee bit nonplused to discover in this manual a scheme or two that he thought he had invented himself, 'way out in California, only to learn that it was an old story back in Birmingham.

Random examples of topics include such diverse items as the partial self-service storeroom, special surface coating for brass laboratory valves, merits of steel versus wood in shelf design, "bulk solutions service," plastic substitute for spiderweb crosshairs, labeling of polythene vessels, electric hazards at different voltages, and training for laboratory management. The chemistry department head on either side of the Atlantic may well get a copy of this informative compendium to pass around to his administrative assistant, laboratory manager, account clerk, chief storekeeper, solutions technician, shop foreman, and instrumental technician, whatever number of persons that may be.

On the debit side, an American laboratory manager will find a few questionable practices or omissions. For example, carbon tetrachloride to fight fire will not meet general approval. Use of self-closing safety cans for flammable solvents, fire walls separating different classes of combustibles, and special techniques for safe recanning of solvents purchased in drums do not seem to have much attention overseas.

The minor sections on "building layout" and "design" are fine as far as they go, but it is hopeless to cover this sort of ground in a small volume. Architecture of chemistry laboratories has now become so complex that the mere employment of a single local general practitioner to design a university chemistry building is inadequate. A faculty building committee will need far more information to offer as soon as their new structure is authorized.

In spite of these minor details, this volume will be interesting reading, especially to the western manager who may be surprised to find how many differences there are in the laboratory vernacular of two peoples who supposedly speak the same language.

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Organometallic Chemistry

Edited by *H. Zeiss*, Monsanto Chemical Company, Dayton, Ohio. ACS Monograph No. 147. Reinhold Publishing Corp., New York, 1960. xiv + 549 pp. Figs. and tables. 16 × 23.5 cm. $17.50.

Reviewers for THIS JOURNAL are urged to try to protect its readers from overenthusiasm on the part of publishers or advertisers, and to judge the effectiveness of a new book as a reference or as a textbook.

In keeping with that responsibility, it should be pointed out that this is not "the first major work on organometallic chemistry in over a decade," as claimed on the jacket. Neither is it a comprehensive survey of organometallic chemistry as its title would indicate, but rather a book about the latest research on this popular subject, written by those who know most about it.

The 14 authors, all acknowledged experts in their fields, do their jobs exceedingly well. Their surveys of their own work and of all the related research supply a great deal of critical, up-to-date information which is certain to be of value to those who give courses on organometallic chemistry, or intend to include that subject in a more general discussion of organic or inorganic chemistry.

The book begins with a chapter on carbon-metal bonding by J. W. Richardson, a review of modern molecular-orbital theory as it applies to organometallic compounds. Dr. Richardson wisely points out near the end of the chapter that "It is quite another problem to prove by computation alone that one bond is stronger than another," and he is careful to compare his conclusions with experience. He explains satisfactorily why mixed complexes predominate in the organometallic chemistry of the transition metals, and his predictions of likely areas of advancement have been borne out very well by later developments which have appeared since the book was written.

The second chapter (Benzyne Chemistry, by Rolf Huisgen) will be of interest primarily to those who study mechanisms of organic reactions in which metals play a temporary but often vital role. The third chapter (Vinylmetallics, by H. D. Kaesz and F. G. A. Stone) is sometimes wordy, but is packed with fact, even to the point of including seven pages of infrared spectra on pertinent compounds. The fourth chapter (Organoboranes, by H. C. Brown) begins with an engaging and sometimes lyrical account of the discovery of the synthesis of boron alkyls from olefins, sodium borohydride, and aluminum chloride in diglyme. The summary of hydroboration reactions is sometimes repetitive, as on page 156, but the account of the hydroboration reaction and its utilization is well organized and clear.

Karl Ziegler's chapter on organoaluminum compounds, while it suffers a bit from minor difficulties of translation and proofreading, is an admirable and exceedingly comprehensive account of the development, the present application, and the future possibilities of organo-aluminum compounds in the areas of synthesis, catalysis, and determination of structure. Many odd and interesting points, usually missed in a reading of Dr. Ziegler's papers, appear in this chapter. It is notable also that Dr. Ziegler's chapter includes the largest number of references to current work, 382 in number.

Chapter 6 is a comprehensive, well-organized review of organosilylmetallic compounds and their application to the synthesis of organometallic substances by Henry Gilman and H. J. S. Winkler, and Chapter 7 is an exceedingly interesting discussion of cyclopentadienyl metal compounds (with 222 references) by P. L. Paulson. In Chapter 8 the editor of the volume, Harold Zeiss, writes the first complete account of the history of organochromium compounds your reviewer has seen, and used it to good advantage as an introduction to the general subject of arene complexes of the transition metals. The step-by-step solution of the many mysteries which surrounded Hein's work between 1919 and 1940 makes pleasant detective-story reading. There follows a very good account of modern work in the field, with an excellent array of references.

Chapter 9, entitled Transition Metal Alkyls and Aryls, by G. E. Coates and F. Glockling, is a systematic review of the great unfolding of organometallic compounds of the 30 transition elements, those elements which for many years were regarded as being incapable of forming any organic compounds. The last chapter, on metal carbonyls and related compounds by J. Chatt, P. L. Paulson and L. M. Venanzi, has in many ways the most difficult assignment of the book. It is handled exceedingly well, with good organization and a text that makes sense from paragraph to paragraph. It appears that these three authors have made the best use of the theoretical development of Chapter 1, using it to explain what otherwise would be an almost hopeless array of strange facts.

As a specialized reference work for those who want to know about the very latest developments in organometallic chemistry, written by those who should know best how to write such an account, this book is highly recommended.

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Ultrafiltration

L. Ambard, University of Strasbourg, France, and *S. Trautmann*, Centre National de la Recherche Scientifique, France. Charles C Thomas, Springfield, Illinois, 1960. x + 67 pp. Figs. and tables. 16 × 23.5 cm. $4.50.

The subject of this small volume is an intriguing one and the physical make-up of the book is most attractive. The foreword by Professor Monnier is a cordial invitation to proceed; appreciation by an unprejudiced reader is promised.

Often in the monograph reference is made to the volume change of hydrated ions, with ionic hydration increasing in dilute solution. Seen in relation to biological systems which contain proteins and electrolytes, ionic hydration is described as having far-reaching implications and permitting the formulation of new (we may say unorthodox) views on the *p*H of body fluids and the real or apparent nature of the cell membrane.

Our comments ought of necessity have to do only with the more physical-chemical items, some of which are collected to form the section, variation in the hydration of ions and constancy of hydration of non-electrolytes explain many phenomena. A few quotations are suggestive of the general approach. Page 32. In connection with the salting-in of globulins: "If the base is NaOH, the neutral salt reduces the hydration of the Na^+ and OH^- ions and consequently renders them more mobile. The mobility of these ion pairs being increased, their collisions with the globulin molecules are more frequent, and they thus recharge the globulins with base. This finally causes the precipitated globulin to redissolve." Page 33. "... a new chapter was added in the text-books of physical chemistry: 'activity of ions' and 'coefficient of activity' of ions. Authors admitted only in the face of irrefutable observations that the activity of a given ion was capable of varying under different experimental conditions. ... But it does not explain why the activity of ions varies; we, for our part, have always been surprised by the way in which many authors have approached this problem ... we have considered ionic activity only from the kinetic point of view."

We hesitate to commend the Ambard-Trautmann monograph to readers of the JOURNAL OF CHEMICAL EDUCATION.

J. W. WILLIAMS
University of Wisconsin
Madison

Name Index of Organic Reactions

J. E. Gowan and *T. S. Wheeler*, both of University College, Dublin, Ireland. 2nd ed. Interscience Publishers, Inc., New York, 1960. vii + 293 pp. 15 × 22 cm. $8.50.

One of the major difficulties in reading the literature of organic chemistry is the predilection of organic chemists to identify reactions, reagents, theories, parameters, indeed, almost anything, by persons' names. Since not only familiar, but also rarer entities are often so identified, there has long been a need for a catalogue, particularly of named reactions. Professors Gowan and Wheeler have so well met this need in their book that it will form a valuable addition to the reference shelf of not only organic chemists, but also industrial chemists and others who must make frequent or occasional use of the organic chemical literature.

The book is a catalogue of 739 named reactions and syntheses, with references to the early published work on the reactions and their major modifications. Also included are a few rules of reaction, such as the Blanc rule (called the Blanc reaction). It is a second, greatly expanded edition. The first, 1950, edition, published by the Society of Chemical Industry, was less widely distributed.

To keep the book within practical limits, the authors have excluded named analytical and color reactions. They showed admirable restraint in excluding all but occasional superficial treatment of reaction mechanisms. Equations with graphic formulas are given for all reactions. The cross reference style, in which all names associated with the included reactions are listed alphabetically, adds to the usefulness of the book. Also useful are the inclusion of a list of allied named reactions with each entry, and a type of reaction index.

In a book as comprehensive as this, one may wonder at the occasional omission of such familiar reactions as the Berthelot hydrogen iodide reduction (now little used, but so also is the Wöhler synthesis), the Dow aniline synthesis, and the Hibbert dehydration method. Nonetheless, the paucity of such omissions is evidence of the general completeness of the treatment.

One may also take exception occasionally to a reaction designation. For example, Grignard reaction is used to name what in the United States is more often termed a Grignard synthesis. Here the Grignard reaction usually refers to the preparation of an alkyl or aryl magnesium halide (which is omitted from the Name Index). However, the authors' inclusion in the majority of cases of descriptive terms with the reaction name, as "Gabriel preparation of primary amines," should set an example for those organic chemists who persist in naming things.

L. OLIVER SMITH, JR.
Valparaiso University
Valparaiso, Indiana

The Science Study Series. Volumes 12 and 18

Doubleday & Co., Inc., Garden City, New York, 1960 and 1961. Available to secondary school students and teachers through Wesleyan University Press, Columbus 16, Ohio. Figs. and tables. Paperbound. Volume 12, 198 pp., $0.95; Volume 18, 195 pp., $0.95.

"The Restless Atom" by Alfred Romer (Volume 12) is a fascinating mystery story. Radioactivity is the plot, but the hero is the physicist-chemist-detective who appears as a composite of all the great names of early 20th century physics. In one sense, this book is a biography of a stirring era in physics. It is more than that, however, in spite of its careful attention to chronology. The human story is skillfully interwoven. The frustrations as well as the inspirations in the laboratory are revealed with delightful candor. The result is one of the best book available to make clear that discoveries are made by *men* and *women*, not by "science."

"Water, the Mirror of Science" by Kenneth S. Davis and John Arthur Day (Volume 18) will be disappointing to teachers of chemistry. All of chemistry—from Democritus through phlogiston to Rutherford and Bohr and eventually to Hoyle's cosmology—gets into the act along with large amounts of physics and some geology. This is definitely not a book for a beginner in science. If he can see the "aims, methods and outlook of science" in this story as is claimed, our guess is that he already has his Ph.D! He certainly can get his facts straight about water better by reading a good modern introductory chemistry text.

W. F. K.

Chemistry of Carbon Compounds. Volume 4. Part C: Heterocyclic Compounds

Edited by *E. H. Rodd*. Elsevier Publishing Co. Sole distributors for the USA, D. Van Nostrand Co., Inc., Princeton, New Jersey, 1960. xviii + 737 pp. 63 tables. 16 × 23 cm. $26.50.

Volume 4C of E. H. Rodd's "Chemistry of Carbon Compounds" concludes the survey of heterocyclic compounds begun four years ago in Volumes 4A and 4B. The 14 chapters in the present text commence with a survey of 6-membered rings with 2 hetero-atoms and continue with a discussion of the chemistry of compounds containing 6-membered rings with more than 2 hetero-atoms, 7-membered and larger rings with various hetero-atoms, purines and related ring systems, nucleosides, nucleotides, nucleic acids, pteridines, alloxazines, and B-vitamins and related compounds. The final 7 chapters (some 332 pages) survey the various alkoloids derived from different ring systems as well as from acyclic systems. The organization, emphasis and style throughout the book remain similar to that found in earlier volumes, and the high quality and lucidity of the predecessors remain in evidence. An index of 69 pages containing approximately 6500 entries referring only to Volume 4C completes the text. Page numbering is consecutive from Volume 4B. Twenty-eight new periodicals are referred to in the present volume, in addition to the approximately 240 periodicals surveyed for the contents of the previous Volume 4B. A staff of 14 authors, including the editor, Professor Rodd, has contributed to the writing of the present volume, which once again, is remarkable in its stylistic homogeneity and uniformity. An errata list of only 14 corrections to previous Volumes 2B, 4A, and 4B again offers testimony to the careful editorial super-

(*Continued on page A840*)

BOOKS FROM INTERSCIENCE

An Introduction to the Chemistry of Heterocyclic Compounds

by R. M. Acheson, *Oxford*

This text for the graduate or senior undergraduate level will be welcomed by teaching organic chemists who, in the past, have often had to rely upon special lectures to introduce this complex subject. It is a concise account of the more important properties and chemical reactions of basic heterocyclic systems, using modern electronic systems and mechanistic concepts where possible. *"In this book Dr. Acheson has succeeded admirably in providing a concise yet highly informative introduction to the chemistry of the fundamental heterocyclic systems without seriously sacrificing important detail. In addition, interesting highlights of many fascinating biochemical areas of the subject are also presented."*—Journal of Chemical Education

1960
356 pages $5.00

Advanced Inorganic Chemistry—A Comprehensive Course

by F. Albert Cotton, *Massachusetts Institute of Technology, and* **G. Wilkinson,** *Imperial College of Science and Technology, University of London.*

Designed expressly for senior and graduate courses in inorganic chemistry, this text will fill the need for a comprehensive presentation incorporating new chemical developments and particularly the recent theoretical advances in the interpretation of bonding and reactivity in inorganic compounds. The book will encompass the chemistry of all the chemical elements and their compounds, introducing interpretive discussion in the light of the latest advances in structural chemistry, general valence theory and, in particular, ligand field theory. There is added emphasis throughout on the pictorial representation of orbitals and bond formation processes. The authors have also prepared an abridgment of this book for use in more modest courses in inorganic chemistry; the "shorter course" will contain some 400 pages and will be available in 1963.

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In press

The Physical Chemistry of Surfaces

by A. W. Adamson, *University of Southern California*

A textbook which develops an understanding of the various manifestations of surface phenomena by a logical progression from fundamental physicochemical principles. The earlier chapters develop the theory systematically and contain problems designed to require critical thinking, not the mere substitution into given equations. The later chapters apply the theory to problems of friction and lubrication; wetting; flotation; detergency; emulsions and foams; chemisorption and catalysis. In these, student reports involving some aspects of the literature take the place of problem assignments. *"It is virtually certain to be a vital force in education and professional circles as surface chemistry comes of age. Adamson is to be congratulated for the service he has rendered."*—Chemical and Engineering News

1960
642 pages $12.75

Quantum Chemistry: Methods and Applications

by R. Daudel, R. Lefebvre and C. Moser, *Centre de Mécanique Ondulatoire Appliquée du Centre National de la Recherche Scientifique, Paris.*

This advanced text clearly presents the important mechanics and the principles of the calculation of wave functions, and describes the methods of their application to the study of chemical reactivity. Recent theoretical and practical developments are included in the presentation. *"All in all, this book certainly represents the most useful and easily assimilated discussion of quantum chemistry and its applications in this field which is available to date. The authors are to be congratulated."*—Science.

1960
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BOOK REVIEWS

vision of this and the previous volumes in this comprehensive treaties.

As Rodd's series on "Chemistry of Carbon Compounds" goes into its final phase (only Volume 5—"Miscellaneous and General Index"—remains to be published), the practicing organic chemist must share the happy amazement of this reviewer that such a complete, well-edited, thoroughly documented and up-to-date series of reference books (nine to date) could have been written and published within the short span of 10 years since the appearance of Volume 1A. Professor Rodd indeed deserves the gratitude of the entire chemical world for the prompt and excellent accomplishment of the Herculean task he set for himself and his collaborators at the outset of this venture.

WILLIAM A. BONNER
Stanford University
Stanford, California

Preparative Methods of Polymer Chemistry

Wayne Sorenson and *Tod W. Campbell*, E. I. du Pont de Nemours & Company, Inc. Interscience Publishers, Inc., New York, 1961. viii + 337 pp. Figs. and tables. 16 × 23.5 cm. $10.50.

Anyone who has been concerned with the synthesis of polymers for teaching purposes will be aware that it is rather difficult to obtain concise, complete directions for such laboratory preparations. At a minimum it is necessary to locate the directions in the original literature. Frequently further details must be added to these before the method is useful to a student. This book is an attempt to remedy the situation by bringing together syntheses of a large number of polymers in sufficient practical detail to be useful. The attempt has been quite successful.

After a brief, useful discussion of general techniques of polymerization, polymer characterization and fabrication, 275 recipes for the preparation of polymers are listed. The recipes are organized into the areas of: polycondensation polymerization, addition polymerization by free radical, anionic, cationic and coordination catalysis, ring opening polymerization, nonclassical polymerizations and synthetic resin formations. An extensive index allows for tracing a given monomer through the various ways in which it may be polymerized, locating intermediates, specific catalysts, etc. Although this major part of the book is essentially a cookbook it is an interesting one to browse through because the recipes are interspersed with short paragraphs presenting interesting aspects of the substance or method under consideration. For example, a random opening of the book has just brought forth a brief discussion of the transformation of ordinary acrylonitrile fibers to black, fire-proof fibers by controlled heating in

(*Continued on page A842*)

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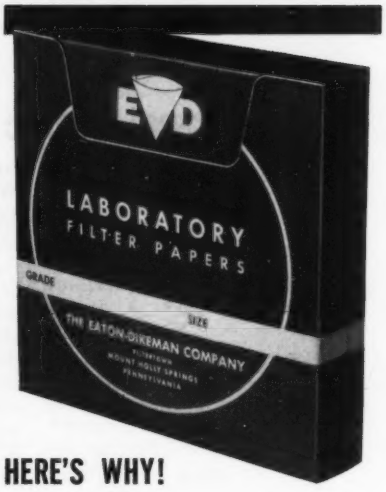

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April, 1962

ORGANIC CHEMISTRY LABORATORY OPERATIONS

By William B. Renfrow and Peter J. Hawkins, both of Oberlin College

This paperbound collection of experiments in organic chemistry stresses modern research methods. The exercises illustrate the techniques and basic principles of laboratory work, and are designed to accompany any standard text in the field. Qualitative and physical organic chemistry, and the design of experimental procedures receive particular attention. There is more material on qualitative organic analysis than in any comparable manual. The experiments have all been thoroughly tested under actual teaching conditions.

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SEMIMICRO LABORATORY EXERCISES IN GENERAL CHEMISTRY, Third Edition

By J. Austin Burrows and Paul Arthur, both of Oklahoma State University, and Otto M. Smith, Kansas State Teacher's College

This outstanding laboratory manual for first year chemistry courses utilizes the more efficient and economical semimicro techniques for both quantitative and descriptive experiments. There are experiments in radioisotopes and modern applications of ion exchange, resins, paper chromatography, determination of ionization constants, and argentimetric titrations. Each experiment is accompanied by carefully prepared practice problems, tables of essential information and materials, and a complete list of reagents and necessary apparatus. The experiments can be used for both two and three-hour laboratory periods. *February, 1962*

LABORATORY PRACTICE OF ORGANIC CHEMISTRY, Fourth Edition

By G. Ross Robertson and Thomas Jacobs, both of the University of California (Los Angeles)

The distinctive feature of this work is its extensive but informal treatment of the principles underlying laboratory practice. It encourages independent scientific thinking by emphasizing analytical experimentation and the preparation of chemical products in finished form. A "Library Problem" will assist the student in becoming aware of the writing skills necessary in the study of chemistry. The "Special Experiments" have been expanded, providing significant special problems for the second semester of a year course.

April, 1962

TEXTBOOK OF INORGANIC CHEMISTRY

By S. Young Tyree, University of North Carolina, and Kerro Knox, Bell Telephone Laboratories

Designed expressly for beginning courses in inorganic chemistry, this text requires no previous background in physical chemistry. Its overall view of the field highlights patterns of behavior among the elements. The material is organized according to groups of elements in the periodic table. For each element, the authors outline occurrence, history, preparation, physical properties, chemical properties and compounds. The new systematic nomenclature is employed throughout the text.

1961, 434 pages, $7.00

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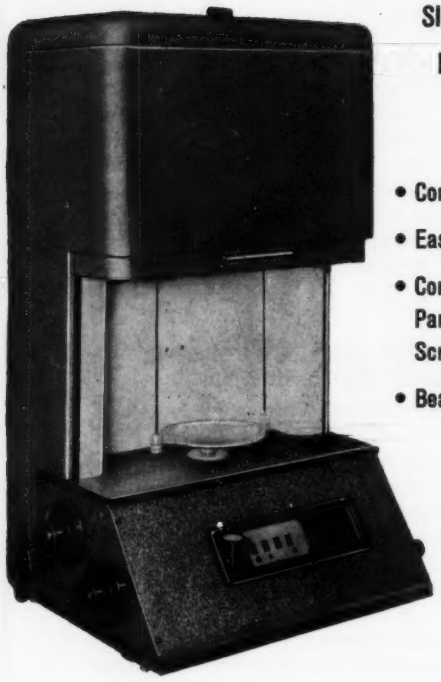

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BOOK REVIEWS

air. Conditions of heating are given and a mechanism for the reaction is presented. Other reactions for the modifications of the polymers of: vinyl chloride, vinyl acetate, acrylonitrile, and dichlorobutadiene are given in the appropriate places.

The coverage of the field is extensive and quite complete. The recipes in general are straight forward and should be easy to follow. Special precautions as to safety and sensitivity of reactants to impurities are well covered. It should be possible for a college student or technician to get a good yield of polymer after only one to two attempts.

This book will make a useful addition to the library of any one interested in making polymers in small batches.

THOMAS FERINGTON
College of Wooster
Wooster, Ohio

Treatise on Analytical Chemistry. Part 1, Theory and Practice, Volume 2

I. M. Kolthoff, University of Minnesota, Minneapolis, and *Philip J. Elving*, University of Michigan, Ann Arbor. Interscience Publishers, Inc., New York, 1961. xx + 497 pp. Figs. and tables. 16.5 × 24 cm. $16.

The present volume comprises the second portion of Part I, Theory and Practice. (For a review of Volume 1, see THIS JOURNAL **37**, 108 (1960).) This section alone is expected to consist of about 124 chapters and to be accommodated in 8 or 9 volumes. Part II, Analytical Chemistry of the Elements, and Part III, Analysis of Industrial Products, will also consist of many chapters and several volumes. Thus the scope of the treatise is truly awe-inspiring—for after 1308 pages and 30 chapters perhaps one fourth of Part I is now in print.

Volume 2 concludes the discussion of Application of Chemical Principles (Section B) and contains a large portion of Section C, Separation: Principles and Technics. Its 11 chapters and their authors are: Reactive Groups as Reagents: Introduction and Organic Applications (Leo Schubert and Irving May), Reactive Groups as Reagents: Inorganic Applications (May and Schubert), Principles of Separation (Lockhart B. Rogers), The Phase Rule in Analytical Chemistry (L. O. Case), Decomposition and Dissolution of Samples: Inorganic (Hobart H. Willard and Charles D. Rulfs), Decomposition and Dissolution of Samples: Organic (E. C. Dunlop), Mechanical Methods (Harold C. Mattraw and Frederick D. Leipzinger), Diffusion Methods (A. Letcher Jones and Glenn R. Brown), Electromigration and Electrophoresis (John R. Cann), Distillation (Arthur Rose), Vacuum Methods (William S. Horton).

Indexing obviously becomes a problem in a work of this magnitude. It is the present plan to provide an index covering Volumes 1, 2 and 3 of Part I, to be included with the forthcoming Volume 3.

(*Continued on page A844*)

Valuable Books in Chemistry from McGraw-Hill

☐ **STATISTICAL MECHANICS**

By NORMAN DAVIDSON, California Institute of Technology. Mc-Graw-Hill Series in Advanced Chemistry. *Available January, 1962.*

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☐ **PHYSICAL CHEMISTRY**

By GEORGE H. DUFFEY, South Dakota State College. Available January, 1962.

This text has been prepared as an introduction to physical chemistry for students who have had calculus and have followed the modern chemical-bond approach in their introductory courses. The theoretical side of physical chemistry is emphasized and only enough descriptive and experimental material is presented to support the arguments and to form exercises in applying the theory.

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BOOK REVIEWS

Thereafter each volume of Part I will contain an individual index. After all the volumes of Part I are published, a complete index to all volumes will be issued.

Volume 2 continues to exhibit the editing care which characterized its predecessor. Individual authors were evidently aware of the contents of other related chapters, for there is remarkably little duplication, and frequent reference to other portions of the treatise. In summary, this volume maintains the high standards set in Volume 1.

JOHN R. HAYES
The Pennsylvania State University
University Park

Treatise on Analytical Chemistry. Part 2, Analytical Chemistry of the Elements, Volume 1

I. M. Kolthoff, University of Minnesota, Minneapolis and *Philip J. Elving*, University of Michigan, Ann Arbor. Interscience Publishers, Inc., New York, 1961. xxi + 471 pp. Figs. and tables. 16.5 × 24 cm. $16.

For reviews of earlier volumes of the treatise see THIS JOURNAL, **37**, 108 (1960).

This, the first volume of Part II, Analytical Chemistry of the Elements, contains two chapters of a general nature and four describing the analysis of specific substances. An excellent chapter (33 pages) on inorganic nomenclature by W. C. Fernelius provides an introduction to this part of the treatise. This chapter may profitably be read by analytical chemists, for it must be admitted that even they are always not above reproach in matters of nomenclature. A brief chapter (9 pages) by James I. Hoffmann provides an interesting review of the development of analytical chemistry and gives some sound general precautions in planning an analytical attack. The determination of hydrogen in its free state is covered in a 32 page chapter by H. F. Beeghly. Logically, the determination of water is next considered in the very complete (137 pages) chapter by John Mitchell, Jr. The analytical chemistry of the inert gases (except radon) is detailed in the 92 page chapter by Gerhard A. Cook. The remaining third of the volume is devoted to a discussion (159 pages) of the analytical chemistry of the alkali metals, written by Silve Kallmann.

The organization of the chapters on the analysis of specific substances exhibits the same features noted in the volume of this section released earlier. The individual chapters are well written, and the reader will find the excellent bibliography accompanying each chapter to be very useful. A subject index to the volume is included.

The character and scope of the treatise is by now too well established to require further comment.

JOHN R. HAYES
The Pennsylvania State University
University Park, Pennsylvania

(*Continued on page A846*)

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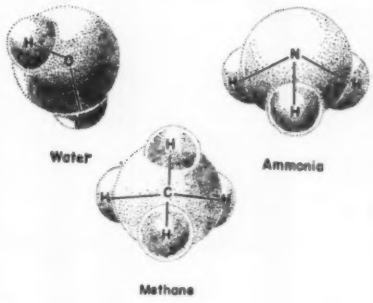

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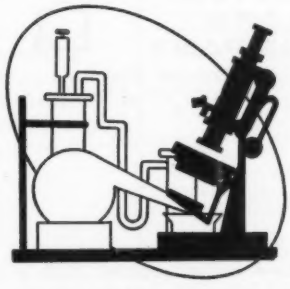

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By GARTH E. LEE, Ph.D., and HARRIS O. VAN ORDEN, Ph.D. 216 pages, 8½" × 11", illustrated. $3.75.

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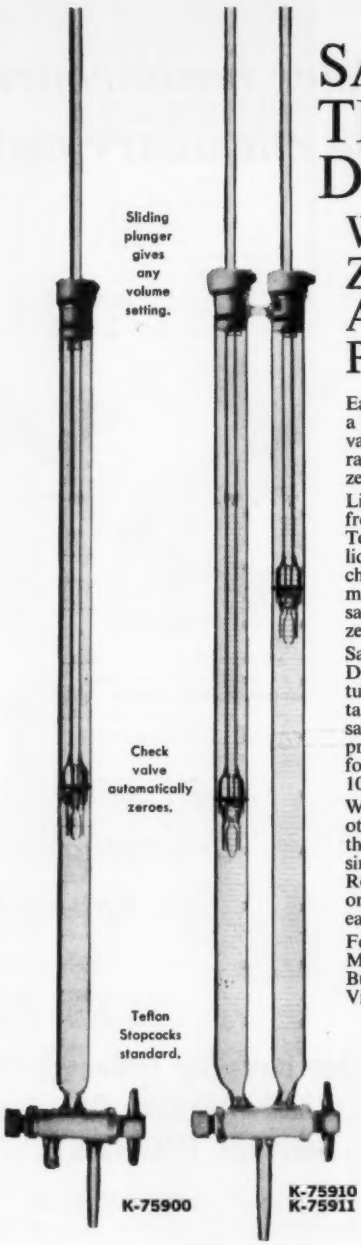

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BOOK REVIEWS

The Nucleic Acids. Volume 3

Edited by *Erwin Chargaff*, Columbia University, New York, New York and *J. N. Davidson*, University of Glasgow, Glasgow, Scotland. Academic Press, Inc., New York, 1960. xvi + 588 pp. Figs. and tables. 16 × 23.5 cm. $18.

A two volume, multi-authored treatise on nucleic acids, edited by Erwin Chargaff and J. N. Davidson, was published six years ago. In line with a prediction by this reviewer these two volumes are now considered as an authoritative work essential to both those who have become participants in some one of the many areas of research involving the nucleic acids as well as those who require background material in order to keep abreast of recent advances. The first two volumes have now been joined by a third.

In the third volume the editors have chosen to provide "a selection of chapters cutting across ... the systemic arrangement observed in the preceding volumes," rather than follow the procedure of revision of earlier chapters. Contributions to the present volume were limited to those who had not collaborated in the preparation of chapters for the preceding volumes. The volume has been composed by its contributors under the following chapter headings: "Deoxyribonucleic Acids as Macromolecules," by C. L. Sadron; "Photochemistry of Nucleic Acids and Their Constituents," by D. Shugar; "Chemical and Enzymic Synthesis of Polynucleotides," by H. Gobind Khorana; "Chemistry of the Nucleic Acids of Microorganisms," by A. N. Belozersky and A. S. Spirin; "The Nucleic Acids of the Bacterial Viruses," by Robert L. Sinsheimer; "The Ribonucleic Acids of Viruses," by Heinz Schuster; "Biosynthesis of Purine Nucleotides," by John M. Buchanan; "Biosynthesis of Pyrimidine Nucleotides," by George W. Crosbie; "The Relationship of Nucleic Acid and Protein Synthesis as Revealed by Studies in Cell-free Systems," by Mahlon B. Hoagland; "Biosynthesis of Proteins in Intact Bacterial Cells," by F. Gros; "Agents Which Influence Nucleic Acid Metabolism" by R. E. Handschumacher and A. D. Welch; and "The Effect of Radiations on Nucleic Acid Metabolism," by L. G. Lajtha.

Each of the chapters is characterized by thorough preparation by individuals with intimate acquaintance with and distinct feelings for the subject matter, yet after reading, one is faced with the uncomfortable realization that a too high percentage of the material has appeared elsewhere, at least once and in some instances several times, in review or symposium format and that the appearance has been too recent to warrant another printing.

Owing to the choice of format for publication certain achievements of lasting nature that have been recorded in the original literature concerning research on nucleic acids for the past six years have been overlooked or mentioned merely in

(*Continued on page A848*)

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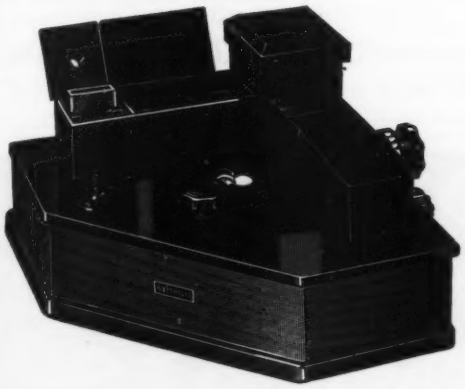

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BOOK REVIEWS

passing. In the opinion of this reviewer the purpose of an authoritative textbook is the evaluation of those achievements which experience seems to indicate to be worthy of special consideration for the less experienced. If this position is accepted, volume 3 should have been composed to provide a better relationship between the amount of revision of older material which should have been undertaken and the shift to newer perspectives.

FRANK WORTHINGTON ALLEN
University of California
School of Medicine
San Francisco, California

Ternary Systems

G. Masing. Translated by B. A. Rogers. Dover Publications, Inc., New York, 1960. Originally published by Reinhold Corp., 1944. v + 173 pp. Figs. and tables. 14 × 21 cm. Paperbound. $1.45.

This is a reproduction of the English translation which appeared in 1944, unaltered except for its cover and price. While written with the metallurgist in mind, the principles which it expounds are those of ternary systems in general, whether metallic or not. The author assumes that the reader already has a good working knowledge of phase equilibria in binary systems. Phase behavior is described for solid-liquid equilibria of many types, including partial and complete miscibility in both the liquid and solid states, and the formation of both congruently and incongruently melting compounds. An introduction on theory is followed by seven chapters illustrating various types of phase behavior and three chapters on specific alloy systems.

The difficulty encountered by students of phase equilibria in understanding ternary systems results from the extra dimension required by the additional composition variable, but the author, an authority in his field, does a commendable job of clearing away the mysteries of the triangular prism with carefully drawn perspective diagrams, sections, and projections. The exposition is clear and the language simple. Frequent page turning is avoided by the repetition of diagrams. The description of actual systems is, understandably, postponed until the simpler, more idealized types are described, since most actual systems, especially with metals, combine many complexities at once. By intelligent paraphrasing (e.g. replacing the words 'solid' by 'liquid', 'liquid' by 'vapor', 'melting' by 'boiling', 'crystallization' by 'condensation', etc.) Chapters 1, 2, 3, 5, 6, and 8 may all be read with profit by those who wish to learn of the phase relations of liquid-vapor equilibria, because of the extensive but often overlooked analogies between melting and vaporization, and the consequent identity of diagram types.

As stated by an earlier reviewer (see THIS JOURNAL, 22, 204 (1945)) more

(*Continued on page A850*)

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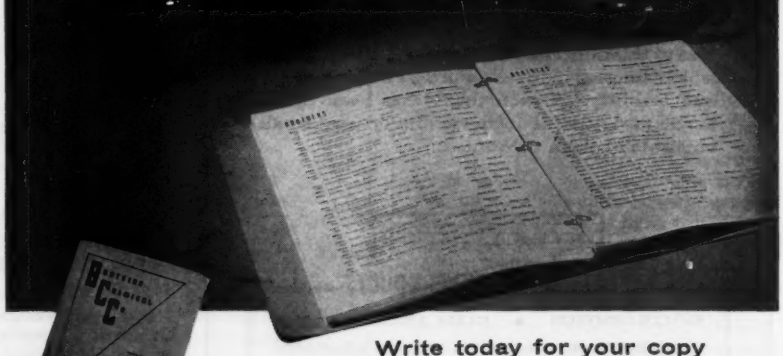

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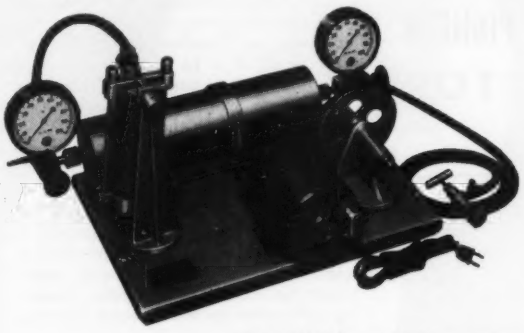

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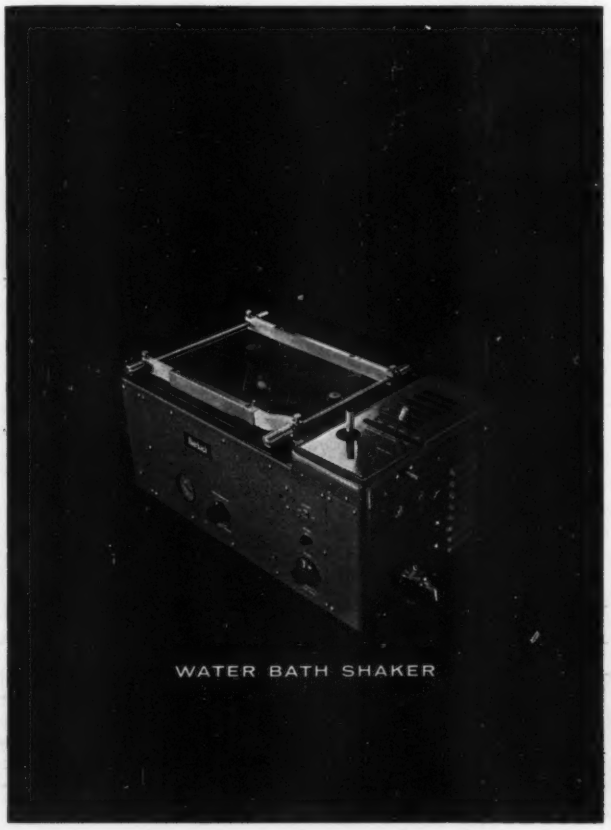

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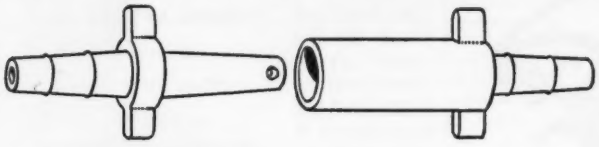

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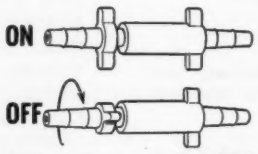

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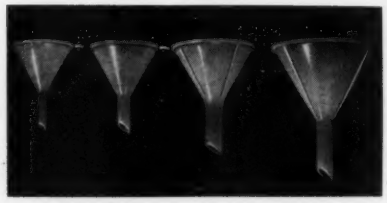

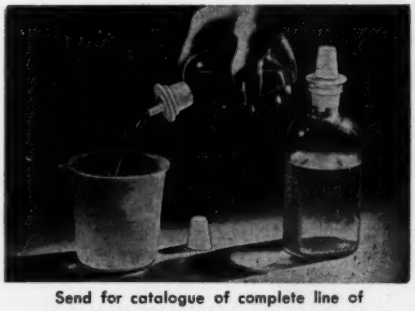

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BOOK REVIEWS

might have been said of the impact on ternary behavior of the presence of an incongruently melting compound in one of the binary systems. It may be pointed out that the term 'monotectic' (p. 107) to describe an invariant equilibrium of the type $L_1 \rightleftarrows S + L_2$ in a binary system, is contrary to the usage of certain other investigators, who apply the term to a binary eutectic in which the invariant liquid has virtually the same composition as one of the two solid phases.

Anyone interested in gaining an understanding of ternary phase equilibria will do well to acquire this standard work, newly available at about one-third of the original price in this colorful paperback printing. One can hardly blame the publisher for including some ten pages of advertising!

NORMAN O. SMITH
Fordham University
New York, New York

Modern Chemical Processes. Volume 6

Editors of Industrial and Engineering Chemistry. Reinhold Publishing Corp., New York, 1961. 126 pp. Figs. and tables. 21 × 29 cm. $6.

Current and reliable process information is often difficult to find; yet such information is most helpful in student training and in making better instructors. The series of papers authored by I/EC editors in collaboration with industrial representatives has aided significantly in providing such information. Volume 6 is the collection of those 17 papers originally published in 1958 and 1959 issues of Industrial and Engineering Chemistry. The present volume continues in the excellent tradition established by earlier ones.

The I/EC editors are to be congratulated for choosing a wide variety of processes, although the emphasis is perhaps too strong on organic technology. Many of the 11 organic processes can be roughly categorized as petrochemical, but three involve use of wood, cotton, and fat derivatives. Only three inorganic processes are described, and three others involve both organic and inorganic chemistry (the preparation of silicones, Grignard reagents, and phosphate insecticides). Flowsheet and operating conditions are reported for each process. Several papers can perhaps, at first thought, be criticized for failure to present more details. Further thought by the reader will probably convince him that he should be thankful that this much information was published about the preparation of a highly competitive chemical.

Process details for preparation of two types of catalysts are most welcome. It is gratifying that two processes are reported for manufacturing relatively small amounts of specialty products. Excellent discussions are given in several papers about plant location, integration of process within plant and company, required flexibility and safety of operation, and eco-

(*Continued on page A852*)

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nomic factors. The book should find wide usage as a reference book. It is highly recommended.

LYLE F. ALBRIGHT
Purdue University
Lafayette, Indiana

The Golden Book of Chemistry Experiments

Robert Brent. Golden Press, Inc., New York, 1960. 112 pp. Illustrated. 21 × 28 cm. $1.95.

This volume should serve as an adequate guide for the youngster who wishes to set up his home laboratory and perform his own chemical experiments. The experiments have been well selected; directions worked out with due regard for safety, for simplicity of apparatus, and of manipulation. The book is copiously illustrated with artistically done sketches which, in general, clearly indicate how apparatus should be set up and what precautions should be observed.

After introductory sections on the "Importance of Chemistry" and "Chemists of the Past" about a dozen pages are devoted to pictures of some of the glassware and hardware used by the chemist, instructions for setting up the home lab, for good manipulative techniques, and for constructing certain "do-it-yourself" items such as a simple chemical balance.

Because of the level for which it is intended the volume has a highly descriptive flavor which does not convey the impression of being a modern approach to the study of chemistry. The modern periodic table which includes element number 102, Nobelium, the mention of silicones, the inclusion of a picture of a nuclear reactor, all combine to reassure us that the work really is up to date. Some persons will be disappointed to see so little attention to atomic structure, chemical bonding, and their relation to chemical properties. The author has obviously had to make his choices and in the opinion of the reviewer has succeeded rather well at the level for which the book is intended.

Coverage of the field of chemistry is rather comprehensive. Experiments include preparation and properties of gases (5), nonmetals (4), metals (8), also sections on water, solutions, acids, bases, salts, colloids, and perhaps a dozen sections dealing with carbon and organic chemistry. Rarely does discussion of a given substance such as hydrogen, iodine, copper, or the hydrocarbons extend beyond two pages of text and in nearly every instance a major fraction of the two pages is devoted to sketches illustrating apparatus and to experimental details. Even at that, it is interesting to observe how much factual information the author has been able to include in so small a space.

The "scientific approach" includes sections on "Mr. Faraday's Candles" and "You-Scientist." These do in a sense provide an imaginative approach to this subject and even though they appear quite limited in scope and seem to provide most of the answers, again, for youngsters this may very well be a more stimulating approach than a rehash of the steps which constitute "the scientific method."

While safety precautions have for the most part been carefully worked out we hope that junior will use a *small* olive jar (as shown) for collecting hydrogen and that he will hold it in a towel before igniting. If the amateur scientist follows directions he should encounter no difficulty in the preparation of chlorine, hydrogen sulfide, or the small amount of phenol suggested.

All in all this is a carefully done volume with a wealth of descriptive chemistry. This might very well be useful as a sourcebook of chemical experiments in the elementary grades.

DONALD S. ALLEN
State University of New York
College of Education
Albany, New York

Fatty Acids: Their Chemistry, Properties, Production, and Uses. Part 1

Edited by *Klare S. Markley,* International Cooperation Administration, U.S.A. Operation Mission to Brazil, Rio de Janeiro, Brazil. 2nd ed. Interscience Publishers, Inc., New York, 1960. ix + 714 pp. Figs. and tables. 16 × 23.5 cm. $22.50.

The appearance of a second edition of

(*Continued on page A854*)

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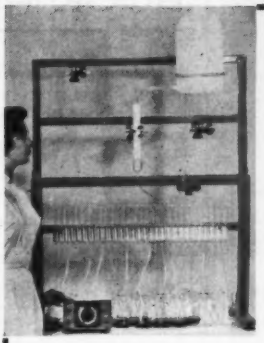

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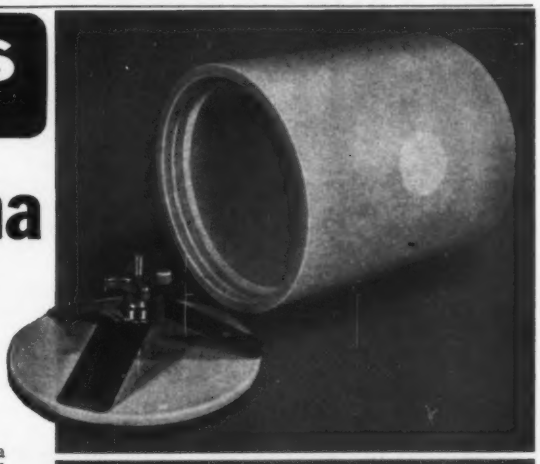

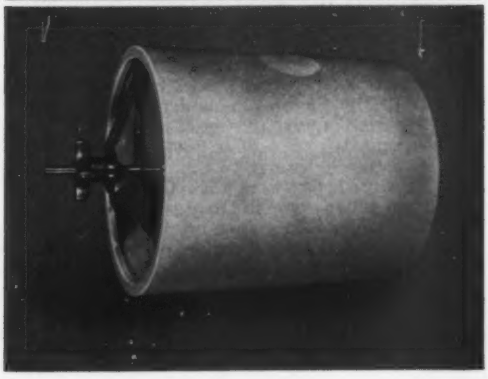

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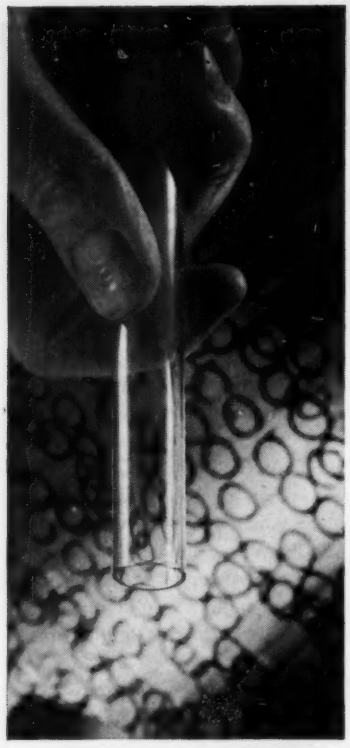

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BOOK REVIEWS

Markley's "Fatty Acids," bringing this important reference work up to date after an interval of 13 years, will be widely welcomed. In the new edition, it has been enlarged from one volume to a promised four, of which the present volume is Part 1. It is also now a cooperative monograph under the editorship of the senior author. Co-authors in Part 1 are Robert T. O'Connor (Chapters 4 and 5) and W. S. Singleton (Chapters 6 and 7).

The scope of the work has been considerably broadened. Types of acids not dealt with directly in the earlier edition—the synthetic odd-numbered acids, the lower members of the fatty acid series, the hydroxy-, keto-, branched-chain, and polycarboxylic acids and their derivatives—are now given equal prominence with the naturally occurring long-chain, normal, monocarboxylic acids. This might appear to be expanding the text beyond the reach of its title, but it does provide a fund of information on these related acids that is not readily available elsewhere. In addition, the preface promises that the industrial production and utilization of fatty acids, which were treated only superficially in the first edition, will be given increased emphasis and be the subjects of added chapters.

The second edition follows the general outline and format of the first. Part 1 contains the following chapters: (1) Historical and General; (2) Nomenclature, Classification, and Description of Individual Acids; (3) Isomerism; (4) X-Ray Diffraction and Polymorphism; (5) Spectral Properties; (6) Properties of the Liquid State; and (7) Solution Properties. In the first edition, these subjects occupy 236 pages of text; here, 673 pages. Their accompanying numbered literature references, previously 407, are now 1571. All the chapters dealing with chemical reactions of the fatty acids, their synthesis, isolation, and identification are left to be taken up in subsequent volumes.

Much of the text of Part 1 has been completely rewritten. Throughout the volume, the relaxing of space limitations is evident in more comprehensive treatment and more effective presentation. The result is a volume of greatly enhanced reference value.

Chapter 2 (227 pp., 678 refs.), as might be expected, received the greatest enlargement. A welcome addition here is the inclusion of descriptions of the acids, mostly omitted in the first edition. Chapter 3 (32 pp., 52 refs.) is kept to its original size, but extensively revised. Chapters 4 (93 pp., 225 refs.) and 5 (120 pp., 362 refs.) are completely reorganized and entirely rewritten. They include an excellent, systematic review of radiation methods and techniques, and they discuss in considerable detail the application of these methods to special problems in the study of fatty acids and related materials. Chapter 6 (109 pp., 145 refs.) which combines Chapters 6 and 8 of the first edition, and Chapter 7 (70 pp., 87 refs.) are revisions in the more customary manner. They modify the wording of the original

(*Continued on page A855*)

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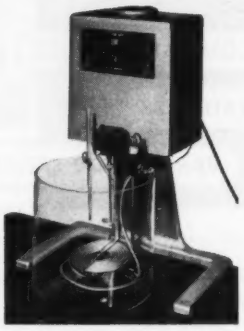

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text only where later information requires it, and insert new paragraphs to cover more recent results. Such additions have enlarged these two chapters by some 50 pages.

It is unfortunate that the large increase in size entails an even larger increase in price. Reference libraries will certainly want the new edition and many chemists, engineers, and technologists will find it important to have a copy conveniently available.

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Reagent Chemicals. American Chemical Society Specifications, 1960

Prepared by the 1960 Committee on Analytical Reagents, ACS. Edited and produced by Applied Publications, ACS, Washington, D. C., 1961. xv + 564 pp. 16.5 × 24 cm.

Much has happened in five years to make the analyst even more grateful to the manufacturers of reagent chemicals. Comparison of this volume with its 1955 older brother makes this increasingly evident. The philosophy of the committee is apparent not only in the clear presentation of the well organized information, but in the space labelled "Notes" at the end of each page. This is a book for the working chemist. It should be available to every student in an analytical course. Perhaps it can cure some of their tendency to take reagents for granted.

W. F. K.

Source Book of the New Plastics Volume 2

Herbert R. Simonds, Consulting Engineer, Stepney, Connecticut. Reinhold Publishing Corp., New York, 1961. x + 310 pp. Figs. and tables. 16.5 × 23.5 cm. $8.95.

Herbert Simonds continues the presentation begun in his "Concise Guide to Plastics" (reviewed in THIS JOURNAL, **35**, 318 (1958)) and continued in Volume 1 of the present series. The properties of recently developed plastics are presented briefly. Brief review articles on current technical processes cover; stereo-specific catalysts and stereo-ordered polymers, block and graft polymers, urethane foams, synthesis and structures, and fluorocarbon nomenclature. This discussion takes up about one-fifth of the book. The remainder is given over to reports from 69 manufacturers on the nature of their most recent products.

The coverage of the plastic field is very complete. This is a good reference book and also a good place to look to get a survey of the applications and potential applications of plastics today.

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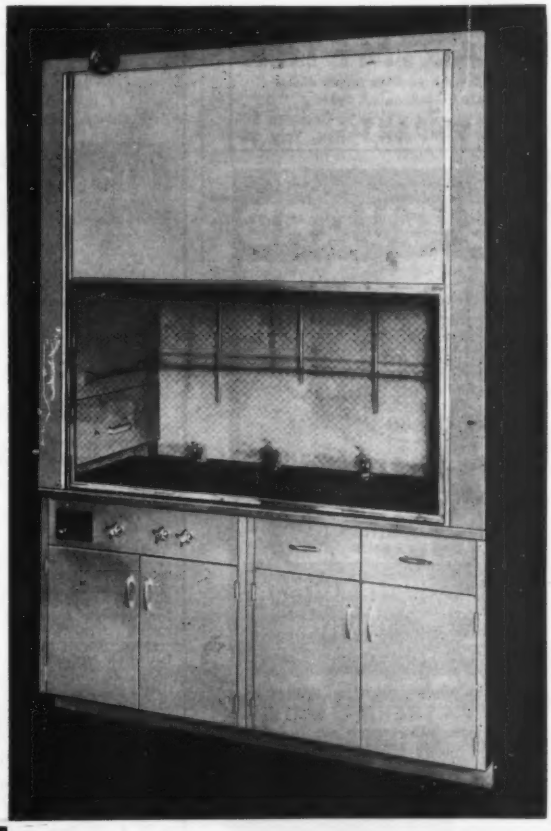

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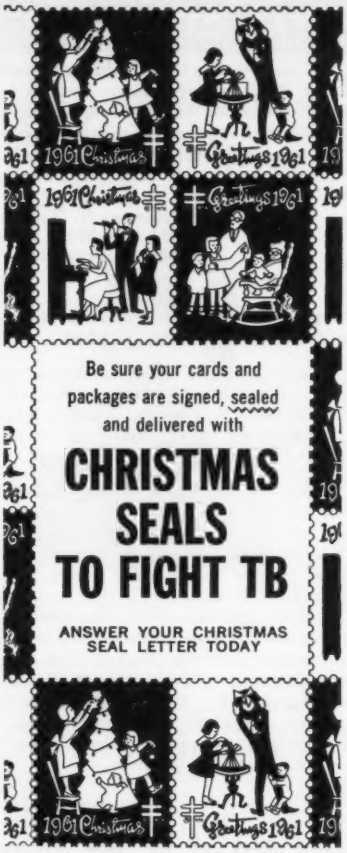

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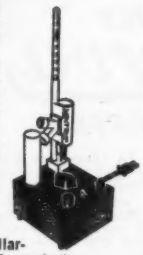

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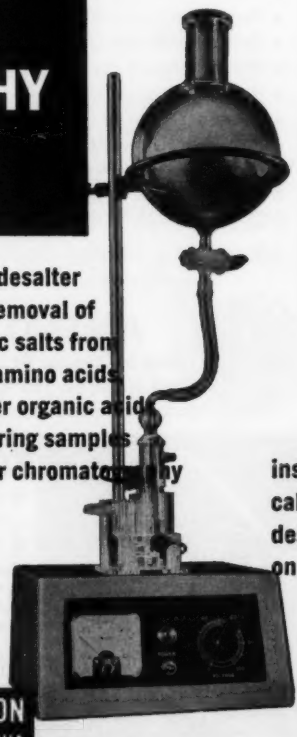

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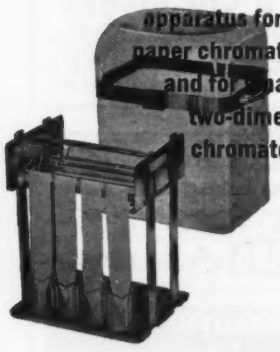

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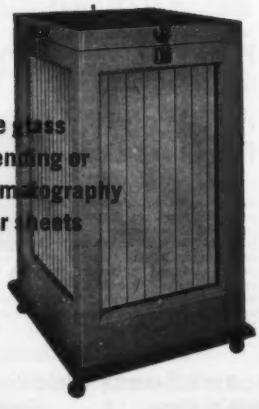

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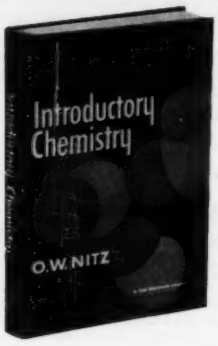

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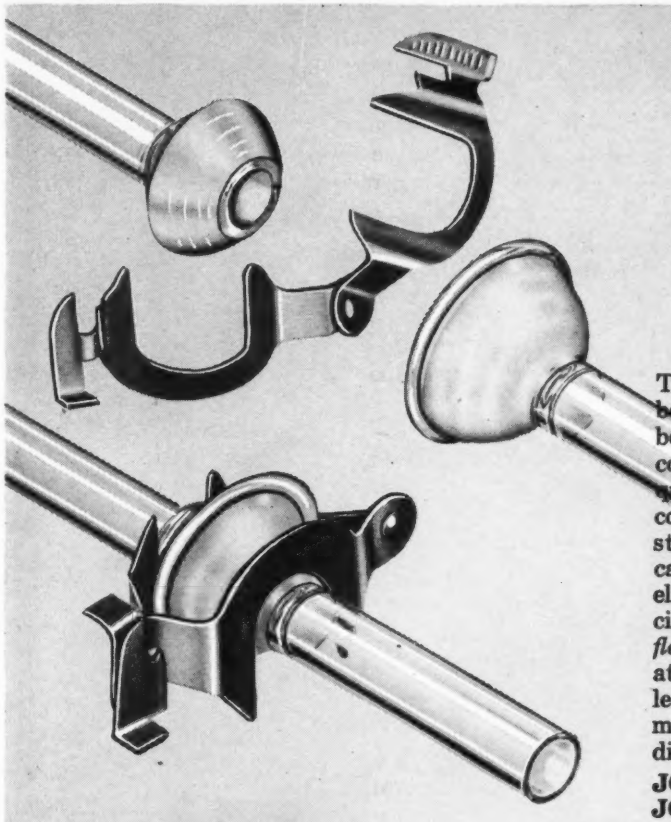

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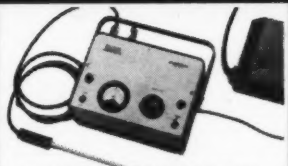

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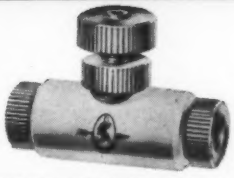

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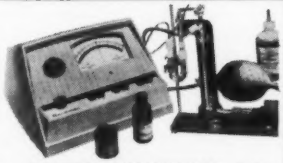

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